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OLIN INDUSTRIES REPORT

on

*DEVELOPMENT OF LECLANCHE
BATTERIES WITH IMPROVED STORAGEABILITY*

to

Squier Signal Corps Laboratories
Contract No. DA-36-039-SC-5489

Final Report

May 1, 1951 to August 31, 1953

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PURPOSE

Contract DA-36-039-SC-5489 between Olin Industries, Inc. and Squier Signal Corps laboratories provides that research investigations shall be conducted leading toward the development of dry batteries that will be capable of long storage at elevated temperatures.

More specifically basic studies are to be conducted on the constituent parts of the LeClanche electrochemical system to determine the characteristics that affect shelf life at high temperatures. These studies shall aim toward production of batteries of the LeClanche type that meet these requirements.

- (a) They shall retain at least 50% of their initial capacity after the following periods of storage under the conditions indicated?
 - (1) 12 months storage at 113°F, 95% R.H.
 - (2) 3 months storage at 130°F, 50% R.H.
 - (3) 2 weeks storage at 160°F, 50% R.H.

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- (b) They shall meet the general requirements of specification JAN-B-18.
- (c) When they are tested at 160°F as specified in JAN-B-18A, par. B-9a the sealing compound used shall not flow.
- (d) Their dimensions shall be such that they can be built into batteries conforming to the standard battery line as proposed by S.C.E.L.
- (e) They shall be so designed that their manufacture shall be commercially feasible for large scale production.

ABSTRACT

This is the final report summarizing the work that has been carried on by Olin Industries, Inc. for Squier Signal Corps Laboratories on investigations leading to the development of LeClanche batteries with improved storageability.

Efforts during the period specified by the contract was directed to the investigation of the:

1. Stability of Manganese Dioxide

Twenty-two types of manganese dioxide of varying crystal structure were stored in battery electrolyte at 113°F, confirming the fact that those materials having an initial structure of epsilon, delta or gamma undergo considerable crystal change when stored in contact with battery electrolyte. Materials having a rho, pyrolusite, gamma B or cryptomelane structure undergo only a slight change in crystal structure under the same conditions.

2. Separator Materials

Forty-three types of separators were tested for a period of twelve months at 113°F. These results show that flours undergo more serious oxidation than most starches. All the starch materials tested to date reduce the voltage of manganese dioxide to about the same level as do corn or potato starch. Generally the reduction of the manganese dioxide (voltage loss) occurs quite rapidly during the first month of storage. Thereafter reduction is relatively light. This is true for all samples tested. Of all the samples tested, none appear to be any more resistant to oxidation than the corn or potato starch.

Thirty-two of the most promising separator materials were tested at 160°F for six weeks, indicating that most conventional starch materials lose their gel structure when in contact with battery electrolyte at this elevated temperature within a period of two to four weeks.

Results have been obtained which show that the majority of the voltage reduction of manganese dioxide when in contact with a starch separator is due to some soluble material present in the initial starch or formed by the acid action of the electrolyte on the starch. Studies have also shown the amount of voltage reduction decreases as the pH of the paste electrolyte is increased until the pH is reached at which diamine forms. After this point, voltage reduction is essentially constant.

3. Polarization

Polarization apparatus was designed to test the passive tendencies of the anode without removing it from the corrosive media.

Considerable work was carried out on anodic and cathodic polarization.

Several types of inhibitors were studied as to their effect on the polarization of sinc. The best inhibitors were found to be Antaron R-155, ammonium dichromate, ammonium chromate, sodium dichromate and a mixture of sodium dichromate and Antaron R-155. However, the chromate film gives high anodic polarization. A desirable inhibitor for a dry cell should cause zinc to exhibit high cathodic polarization and moderate anodic polarization. Amalgamation of zinc does not give satisfactory inhibition, as the amalgamation process tends only to equalize the voltage of anodic and cathodic areas on the zinc surface. It does not set up a corrosion barrier in terms of polarization. This means that any potential difference on the zinc surface will cause serious corrosion. With amalgamated zinc especially, the corrosion process does not appear to be a direct liberation of hydrogen but rather the discharge of free zinc which in turn reacts with the electrolyte to form hydrogen.

Continued polarization studies show that the chromate ion is the best inhibitor tested to date for cells which are stored at elevated temperatures. Cells have been fabricated and tested which gave very satisfactory service after storage at 113°F, 95% R.H. for one year. Best results were obtained using a chromate inhibitor in the paste and barium chromate as a reservoir for chromate ions in the depolarizer itself.

Weight loss measurements and polarization studies indicate that percentages above 1.0% of chromate is the best concentration for inhibiting zinc, expecially at elevated temperatures. These same studies were made with varying degrees of amalgamation of zinc and indications are that this process is not satisfactory for inhibiting zinc at elevated temperatures due to the fact that little or no corrosion barrier is set up.

4. Subseal

Cells using a polyethylene gasket as a substitute for a wax sub-seal, with a heat resistant polystyrene top seal gave better capacities and exhibited considerably less leakage than cells with only a wax subseal and heat resistant polystyrene top seal after storage for two weeks at 160°F.

5. Substitute for Starch-Four Gel Separator

The study of liner materials to replace the conventional starch separator in cells which must undergo elevated temperature storage, including paper liners coated with polyvinyl alcohol, carboxy methyl cellulose, methyl cellulose, proteins, glutens, and gum type materials. Data available to date, indicates that cells fabricated with methyl cellulose coated paper liners, polyethylene gasket as a sub-seal and heat resistant polystyrene top seal, have met the requirements of 130°F, 50% R.H. and 160°F, 50% R.H. storage capacity.

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PART I

EQUIPMENT - PROCEDURE

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FACTUAL DATA

Part I - Equipment and Procedure

1. High Temperature Baths and Humidity Ovens

Four water baths were purchased from the Precision Scientific Company of Chicago, Illinois. Three of these were put into operation, and one was kept in reserve in case of mechanical failure in any one of the others. Two baths have a working area of $18^{\text{m}} \times 12^{\text{m}} \times 8^{\frac{1}{2}^{\text{m}}}$ each and the other two have a working area of $36^{\text{m}} \times 18^{\text{m}} \times 12^{\text{m}}$ each. They are thermostatically controlled and can be maintained within plus or minus one-half of one degree of the desired setting. Also, they are equipped with a device which, besides maintaining a constant water level, will also provide for water circulation and more constant conditions throughout.

These baths were used to store manganese dioxide samples, and to maintain the correct temperature conditions for corrosion experiments, starch-flour voltage reduction of manganese dioxide, storage of samples used for polarization studies and ion substitution into the structure of a gamma type manganese dioxide.

Two constant humidity ovens were ordered and purchased from the American Instrument Company of Silver Springs, Maryland. One steam humidified cabinet, inside dimensions 24" x 24" x 24" stainless steel interior and lacquered steel exterior for operation on 230 volts, 60 cycles A.C. current, one steam humidified cabinet, inside dimensions 50" x 24" x 24" stainless steel interior with lacquered steel exterior for operation on 230 volts, 60 cycles A.C. current.

Completed cells were stored in these ovens at the conditions and for the times specified by this contract. These are two weeks at $160^{\circ}F$ and 50% relative humidity, three months at $130^{\circ}F$ and 50% relative humidity and twelve months at $113^{\circ}F$ and 95% relative humidity.

2. Polarization Apparatus

The polarization apparatus used in this study is shown in Figure 1. It consists essentially of two units. One unit supplies the polarization current and imposes a desired anode current density on the zinc electrode. The power source is a three volt battery connected in series with a variable resistance box, a milliammeter, and a switch. One lead of this circuit is connected to a carbon rod which serves as a power electrode, the other lead to the zinc anode. This anode has a controlled surface area of 0.785 cm. By means of the resistance box it is possible to vary the current in the circuit which in turn controls the current density of the anode. The current density was regulated to such a value as to compare with existing tests on cells of normal construction

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The second unit of this apparatus consists of a zinc anode and the electrode of a saturated calomel half cell. The measurement obtained is the open circuit voltage of the zinc anode against the calomel reference electorde. The initial closed circuit voltage of this couple is imposed on the anode and the voltage can be read at various intervals of time after the polarization current has been started, and while polarization current density is held constant by means of the variable resistance box. A saturated calomel half cell is electrolytically connected with the corrosion solution by means of modified salt bridge which has a saturated potassium chloride solution in the arm contacting the calomel cell. The other arm of the bridge can be filled with corrosion solution under investigation. The junction of the two liquids occurs around a three-way stopcock shown in Figure 1.

The voltage measurements are made by an electronic voltmeter and a potentiometer which are connected in the circuit with a double throw switch. This makes it possible to read the voltage with either the voltmeter or the potentiometer. The former is used to obtain accurate measurements of the open circuit voltage.

A modification of this apparatus is used to measure anodic and cathodic polarization jointly. In the latter tests, a zinc strip of controlled area (0.785 sq. cm.) is substituted for the carbon power electrode in the power supply. The polarization current is varied and readings are obtained from each electrode.

The construction of the zinc anodes is of particular interest, they consists of a zinc disc one centimeter in diameter which is bonded by means of an inert wax (probably DeKotinsky cement) to a short section of glass tubing of the same external diameter. A wire lead extends down through the glass tube and is soldered to the center of the disc. Both the wire lead and the open end of the glass tube form a vapor tight seal with a rubber stopper.

The rubber stopper which fits a sample bottle contains two other holes which accommodate one leg of the salt bridge and the power electrode (See Figure 1). During storage periods these holes are closed with a short section of solid glass rod. It is to be noted that the zinc electrode can be tested on the polarization apparatus without removing it from the corrosion media. Furthermore because of the construction of the salt bridge the corrosion solution is not contaminated by foreign ions during the polarization measurements.

3. Storage Containers for Manganese Dioxide Studies

Different crystal types of manganese dioxide were stored in glass sample bottles in water baths maintained at the temperatures specified by this contract.

4. Electron Microscope

The electron microscope was used for the most part in examining the crystallographic changes occuring in the manganese dioxide during storage.

Additional checks were made by X-ray diffraction whenever it seemed necessary.

5. Chemical Analysis

Chemical analysis was used to determine the amount of reduction of mangamese dioxide caused by separator materials. It is also being used to characterize further new types of manganese dioxide.

6A. Apparatus for Measuring Manganese Dioxide Voltage (Untamped Depolarizer

The voltage of manganese dioxide samples stored in contact with various separating materials was measured by employing the following techniques:

A hole 3/32" in diameter and 1/8" depth is drilled in the end of a carbon rod and a small quantity of mix is solidly tamped into this cavity. The end of the rod containing this miniature mix slug is then pressed against a flat zinc anode using starch coated flat cell paper as a separator. The voltage of the couple is read on a potentiometer or very high resistance voltmeter. The method had proved reliable and reproducible. It is possible also to read the voltage of the manganese dioxide coupled with a calomel reference electrode instead of a zinc electrode by a slight modification in the procedure.

6B. Apparatus for Measuring Manganese Dioxide Voltages (Tamped)

The voltage of manganese dioxide samples stored as AA cores in contact with various separating materials was measured in the following manner:

One leg of a modified salt bridge is filled with electrolyte and brought into contact with the separator material which has been stored in a glass jar in contact with an AA core of the manganese dioxide being tested. The second leg of this salt bridge (containing a saturated KCl solution) contacts a calomel half-cell. A lead wire from the calomel cell to the negative terminal of a potentiometer and a lead wire from the carbon rods of the AA core to the positive terminal of the potentiometer completes the circuit. It is then possible to read the voltage of the manganese dioxide coupled with the voltage of the calomel reference electrode. This procedure eliminates problems which arise when a zinc anode is present.

7. Machine for Coating Paper

A gum label machine was purchased from New Jersey Machine Corporation of Hoboken New Jersey. This pony gummer was adopted for coating short lengths of paper.

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PART II

STABILITY OF

MANGANESE DIOXIDE

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Part II - Stability of Manganese Dioxide

1. Effect of Stability on Various Crystallographic Types of Manganese Dioxide

Under tropical storage conditions, background data indicated that certain types of manganese dioxide, when placed in contact with battery electrolyte, were stable and underwent no crystal phase conversion while other types were unstable. This stability or instability directly influenced capacity on delayed tests, and the problem became even more critical as temperatures rose. This conversion or phase change was not critical in itself, because the new phases, when produced outside the dry cell, performed excellently. However, the new physical shape of the manganese dioxide particles formed in the dry cell was extremely important. This change of shape that took place during conversion caused a loss of contact between the particles of manganese dioxide and carbon black. This loss of contact of course made it impossible for the particles of manganese dioxide to participate in the discharge reaction. First proof of this was obtained by re-working the mix from cells in which conversion had taken place, thus re-establishing contact between manganese dioxide and carbon black so that capacities were as high, if not higher, than the original cells.

With this knowledge, a study was initiated in which twenty-two samples of manganese dioxide from various sources and covering the whole gamut of crystallographic types were analyzed.

This was accomplished by placing the manganese dioxide in normal battery electrolyte (24% NH₁Cl, 22.5% ZnCl₂, 53.5% H₂O composition by weight) for storage in such a manner as to approximate conventional battery conditions. Each sample of manganese dioxide was stored as a mix consisting of seven parts ore to one part Shawinigan black in contact with battery electrolyte in sealed glass jars. The samples in these jars were then stored in water baths at temperatures of 113°F., 130°F., and 160°F. Samples at 130°F. and 160°F. were later eliminated due to malfunction of the water baths; however, limited results were obtained which showed that little significant difference occurred at these higher temperatures. It was noted that the crystal structure change occurred more rapidly at 160°F. as would be expected due to increased chemical activity.

Small amounts of each sample were removed at various intervals during the storage period and examined with an electron microscope to determine diffraction patterns and morphological structure. These results are compared to analyses of the initial material to show just changes have occurred.

The results of this work are tabulated in Figure #1 and the condensed results are illustrated in a phase diagram, Figure #2.

The summary of the results are as follows:

Delta Materials - Highly unstable and tend to change to epsilon and then to alpha or convert directly to alpha.

Epsilon Materials - Highly unstable and tend to convert to alpha.

Alpha Materials - Extremely stable.

Rho Materials - Extremely stable.

Beta Materials - Extremely stable.

Gamma A Materials - Tend to convert to alpha. The alpha product, however, is different in appearance than that formed by delta or epsilon conversion. It is hairlike while the latter is bar or burrlike.

Gamma B Materials - If pure phase, appear extremely stable; however, if a Gamma A phase or alpha is present, the stability is lost.

Some gamma materials appear to convert quite rapidly while others do not show a conversion tendency. This can be correlated with the difference in diffraction patterns of various gamma material. For simplicity, the gamma classification has been broken down to include a gamma A and a gamma B subdivision. These sub-divisions are arbitrary and are based upon the relative intensities of the 1.38 A° and 1.44 A° lines of the conventional gamma diffraction pattern. Gamma A material is defined by a diffraction pattern containing the 2.4 A°, 2.1 A°, 1.62 A° and 1.44 A° lines, while gamma B is defined by a diffraction pattern containing the 2.4 A°, 2.1 A°, 1.62 A° and 1.38 A° lines. It is possible and quite common to have a poly-phase gamma material wherein the material assumes the stable characteristics of gamma A.

Of some note is the fact that the major structural change of samples tested took place during the first two months of storage. This answers in part the question of poor delayed performance of many materials when tested at three months time along with the effect of pH change, starch reduction and breakdown of cell efficiency.

Capacity results have indicated that rho and beta phase material, although extremely stable, are far from outstanding performance-wise. Whereas alpha and pure phase gamma B not only are stable but also excellent in performance.

To illustrate further the phase changes of an unstable manganese dioxide, work was done using an ore of delta structure (light hydrate). When stored in battery electrolyte for extended periods, light hydrate converts from an apparently unstable delta pattern, through an epsilon stage to an apparently stable cryptomelane phase. With this in mind, experiments have been carried out in which light hydrate was converted to cryptomelane (alpha) before being assembled into batteries. These experiments consisted of leaching light hydrate in different solutions at a constant temperature of 90°C for seventy-

two hours. The solutions tested were 20% ZnCl₂, 20% NH₁Cl, 20% KCl and H₂O. In all cases, a pH level between 3.5 and 4.0 was maintained by the addition of dilute HCl. Samples of the product were removed at intervals of twenty-four hours to determine the rate at which conversion was taking place. After seventy-two hours at 90°C the solutions were cooled, filtered, washed and then dried in a forced draft oven for twenty-four hours at 90°C.

Besides taking electron diffraction patterns and micrographs of the samples, a chemical analysis was run on the final product. Results of this experiment are given in Figure #3. Except for one case, the final products of leaching gave cryptomelane diffraction patterns. This one exception being KCl solution were conversion was not completed in seventy-two hours.

The chemical analysis shows that the ZnCl₂ cryptomelane has a density much above that of the others and a formula more close to MnO₂ (MnO_{1.96}). These factors tend to prove that the ZnCl₂ cryptomelane is the best of the materials for good storage characteristics. These results are substantiated by capacity results in Figure #4.

2. A Study of the Effects of Introducing Various Ions into the Structure of an Essentially Gamma Type Manganese Dioxide

A study was initiated to determine the effect on the crystal structure of a gamma type manganese dioxide by the introduction of various ions into its structure. Electro-chemical Company was chosen as the manganese dioxide due to its structure which was typical for electrolytic ore. The following salts were introduced into the structure of this ore:

Sodium Chloride
Sodium Sulfate
Potassium Chloride
Potassium Sulfate
#Barium Chloride
#Barium Sulfate
Calcium Chloride
Calcium Sulfate
Manganese Chloride
Manganese Sulfate
Ferric Chloride
Perric Sulfate

Copper Chloride
Copper Sulfate
Lead Chloride
Lead Sulfate
Zinc Chloride
Zinc Sulfate
Magnesium Chloride
Magnesium Sulfate
Lithium Chloride
Lithium Chloride
Lithium Sulfate
Ammonium Chloride
Ammonium Sulfate

In the samples tested, one hundred grams of manganese dioxide plus two hundred grams of distilled water were used and an acid added (H2SO4 for sulfate salts and HCl for chloride salts with content varied from zero to thirty-

* Barium salts were discarded due to inability to wash the product thoroughly and to achieve consistent capacity results.

five grams). The weight of salt added was half the molecular weight in grams except in cases where the valence was greater than one, here one-fourth or one-third the molecular weight was used. The amount of salt used was far in excess of that needed to carry out the reaction but it was necessary to use this excess to smother the effect of any other ion present. After the samples were made up, they were stored in a water bath at 90°C. for a period of a week, After storage the converted ores were washed, filtered and dried.

The crystallographic results of this experiment are tabulated in Figure #5.

Curves shown in Figure #6 detail the conversions which take place in the various acid mediums. It must be understood that these curves are based on electron diffraction analysis and therefore are extremely relative. The quantity of any phase present is based on its relative intensity with regard to the other diffraction lines in the pattern. The intensity of each phase is based on a scale from one to ten.

Evaluation of these converted ores was accomplished by the fabricating and testing of AA size cells for capacity results. This size cell was used for the evaluation because of the small amounts of ore available. The tests on which these cells were evaluated were picked to approximate current density drains equivalent for D size cells. The four tests in the series are 25 ohm light industrial and 650 ohm continuous which are low rate discharge tests and 25 ohm heavy industrial and 66.7 ohm continuous which are high rate discharge tests.

Capacity results are given also in Figure #5 but are incomplete due to difficulties in testing and fabricating these cells.

All ores have been fabricated into cells and results are incomplete pending completion of testing.

The following observations in regard to this study are presented:

Ammonium, potassium and lead favor the formation and stabilisation of the alpha phase regardless of acid concentration.

Zinc, calcium and copper favor the formation and stabilisation of the rho phase dependent on acid concentration.

Sodium, magnesium, lithium, manganese favor the formation and stabilisation of the beta phase, dependent upon acid concentration.

Probably the most significant change was introduced by the iron salts which increase the gamma component markedly.

#Exception calcium sulfate

It can be noted that at the highest acid concentration, no samples remained unconverted. Also at no time was there any indication of reduction to Mn_2O_3 or some lower oxide.

Based on the assumption that gamma and rho are highly distorted or strained phases of beta a very logical progression can be illustrated as in Figure #7.

It is highly possible that the inherent strains or distortion in the original crystal were relieved completely, (formation of beta) or partially (formation of rho) or increased (formation of a stronger gamma phase). However, the part that alpha plays in this progression cannot be determined unless the unique form of alpha created is also a member of this family.

Some last minute work has been attempted in a try at converting pure phase beta material to gamma by the use of FeCl₃ and HCl at elevated temperature. This has met with failure. But this failure in itself may be a further answer in that the extremely stable beta phase may be completely lacking in voids or channels in the crystal structure, which we know are present in other structures. This lack may make it impossible for the ferric ion to get a foothold and cause a reverse condition.

CONCLUSIONS

- 1. Delta, epsilon and gamma A types of manganese dioxide are essentially useless as a depolarizer in dry batteries unless converted to a stable phase,
- 2. Gamma B, alpha, rho and pyrolusite or beta are extremely desimable as depolarisers in a dry battery. However, the latter two are rather poor performers.
- 3. The major crystrallographic change in manganese dioxide, if any, will take place within two months in a battery on shelf.
- 4. It is not the conversion material itself that is responsible for poor delayed performance, but rather the inability of it to participate in the discharge reaction due to loss of physical contact with the carbon black.
- 5. Introduction of ions into manganese dioxide plays and important role in its crystallographic make up.
- 6. In converting manganese dioxide by introducing ions, it is possible to control the end product by selective use of ions and acid concentration. In other words, it is possible to increase or decrease the amount of distortion or strain within a crystal.
- 7. It is possible that the whole crystallographic manganese dioxide family is based directly on the beta structure.

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TABLES AND CHARTS

PART II

STABILITY OF MANGANESE DIOXIDE

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of Various Crystal Structures in Contact with a Conventional Battery Electrolyte and Tabulated Results of Studies of Crystallographic Changes of Manganese Dioxides Stored at 113°F. in Glass Jars,

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FIGURE

(Maj.) Par Albha (Trace Gamma-B(Tr. Rho and and Gamma-B months Alpha Epsilon Alpha Beta Alpha Alpha Alpha-Alpha Beta Rho Rho Rho 12 Epsilon(Maj) Epsilon(Maj)Epsilon(Maj) Alpha (Min) Alpha (Min)Alpha (Min) (Ma.j. Albha (Trace Alpha (Maj. Alpha (Maj.) Alpha (Maj. Alpha (Maj. Gamma-A(Min)Gamma-A(Tr. Gamma - A (Min Gamma - A (Min and Samples 9 months Rho and Gamma-B Alpha Klpha Beta Alpha Beta Rho Rho Rho Alpha (Min Alpha of (Maj.) Alpha (Trace and Struc ture 6 months Alpha Garmma -3 Alpha Rho and Alpha Be ta Rho Be ta Rho Rho Alpha (Trace Alpha (Maj.) Alpha (Maj.) (Maj.) Gamma-A(Min) Gamma-A(Min) artj months Rho and Alpha Be ta Crystal Alpha Gamma-B Alpha Rho Beta Rpo Rho N Alpha (Trace (Maj.) Initially and and Alpha Garrma - Æ Gamma-B Epsilon Gamma-A Alpha Beta Be ta Alpha Alpha 阳0 Rho Rho Rho 000 ç ç Baker's Analytical Manganese Dioxide Burgess Chemical Japanese Electro Lavino Type E.C. Synthetic Mn02 Import Chemical New Jersey Zinc Verona Chemical Asbury Chemical National Carbon Synthetic Mn02 Chemical Mn02 Caucasian Ore Artifical Ore R.S. Dean Ore Ore Ore

Cont.	
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		Crystal	tal Structure		
Manganese Dioxide	Initially	2 months	6 months	9 months	12 months
French Erogogene	Rho and Alpha	Rho and Alpha	Rho and Alpha	Rho and Alpha	Rho and Alpha
Chemical MnO2 General Dry Battery Co.	Rho and Alpha	Rho and Alpha	Rho and Alpha	Rho and Alpha	Rho and Alpha
African Ore	Rho	. प्र	Rho	Rho	Rho
Light Hydrate Import Chemical Co.	Delta	Epsilon(Haj. Alpha (Hin)	Epsilon(Maj) Alpha (Min)	Epsilon Par Alpha	Alpha (Maj) Epsilon(Lin)
Montana Ore	Epsilon Alpha	Epsilon Alpha	Epsilon Alpha	Epsilon Alpha	Epsilon Alph
Inoz Lot No. 18 Western Electro Chem.	Gamna-A(Kaj) Gamna-B(Min)	Alpha	Alpha	Alpha	Alpha
Dellite Lot No. 25 E.J. Lavino Co.	Rho (Maj) Alpha (Min)	Rho (Haj) Alpha (Tr.)	Rho (Taj) Alpha (Tr.)	Rho (Kaj Alpha (Fr.	Rho (Yaj) Alpha(Tr.)
Electrolytic MnO2 Bright Star Battery Co	Germe-A(Nej) Germe-B(Tr.)	Alpha (Maj) Gamma-A(Min)	([a])	Alpha (Kaj Carma-2 (Kin	Alpha
Chemical MnO2 Continental Chemical Co	Rho	Rho	Pho	Яйо	Rho
Gosta Rican Ore	Eps11on	Epsilon(Maj) Alpha (Min)	Epsilon) par Alpha	Epsilon Par Alpha	Epsilon Par Alpha
Electrolytic MnO2 Burgess Battery Co.	Ganma – B	. Gamma-B	Gamma – B	Са∴та −В	Garma-B(Ma) Garma-A(Tr.

FIGURE 1 (Cont.

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Crystallographic Progression for the Various Crystal Phases of Manganese Dioxide

FIGURE 2

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FIGURE 3

		rystal Structure		
	Light Hydrate Leach of 3.5 to 4.0. pH	•		ric acid.
,	.Le	aching Solutions	3	
	20% NH4c1	20% KC1	H ₂ 0	20% ZnCl ₂
Initial Structure	D elta	Delta	Delta	Delta
Structure after 24hrs in solution @ 90°C.	Epsilon (major), some delta	Finely divided epsilon (major, some delta	some delta, some epsilon, fine traces cryptomelane	some delta, finely divided ed crypto- melane (maj.
Structure after 48 hr in solution @ 9000.	Epsilon (major), some cryptomeland	Epsilon (major, some delta, very minor cryptomelane		finely divided cryptomelane
Structure after 72 hr in solution @ 90°C.	Cryptomelane (major)	Epsilon (major, some cryptomelane	Cryptomelane (major)	finely divided cryptomelane
	Chemical Analys	is After 72 Hour	es At 90°C.	
Density	4.26 gms./ cu.in.	9.73 gms/cu.in	5.15 gms/cuir	10.44 grams per cu. in.
Mn (dry)	55•9 %	53•3%	55•7%	51.1%
MnO2 (dry)	79•5 %	82.4%	84•2%	78.6%
н20	3.1 %	10.96%	8.36%	4.04%
Formula of resulting ore	Mn0 _{1.88}	MnO1.97	Mn01.95	Mn ⁰ 1.96
orphological haracteris- ics	Bar-like Crystals		Bar-like Crystals	Bar-like Crystals

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FIGURE 1

LeChanche' Type "D" Cells Stored at a Temperature of 160°F.

			!	Test Res	ults	· · · · · · · · · · · · · · · · · · ·	
Book '	Mix Formulation	BA	30	ВА	8	BA 4	08/u
No.		Initial	2 wks.	Initial	2 wks.	Initial	2 wks.
ŧi		(days)	@ 1 60F	(hours)	@ 1 60F	(hours)	@ 160F
	100% Cryptomelane						
B7727	(Light Hydrate)	11.1	1.5	2 1 6	56.4	21.5	1. 7
B3327	leached in H20	ىلە ● بىلىرىك	T.*2	210	70.4	21.0	4.7
	7.25/1- ore/black	dys.	dys.	hrs.	hrs.	hrs.	hrs.
В3330	30% Cryptomelane (Light Hydrate leached in H20) 70% African Ore 7.25/1- ore/black	13.4	No Good	173	48.1	17•7	1.5
B3334	100% Cryptomelane (Light Hydrate leached in KCl sol)	9•9	7-4	1 89	29 <i>•</i> 5	22•0	1.0
B3337	7.25/1- ore/black 30% Cryptomelane (Light Hydrate leached in KCl sol) 70% African Ore 7.25/1- ore/black	13•5	No Good	190	29•0	22•3	4.0
B3342	30% Cryptomelane (Light Hydrate leached in NH ₄ Cl) 70% African Ore 7.25/1- ore/black	12•3	3.1	125	60 .9	15•3	7•00
В3346	100% Cryptomelane (Light Hydrate leached in ZnCl ₂) 7.25/1- ore/black	13.1	4•3	170	78•0 ·	26.0	9•8
B3 3 49	30% Cryptomelane (Light Hydrate leached in ZnCl ₂) 70% African Ore 7.25/1- ore/black	1 5•0	6.4	198	79•0	16.6	9•5

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of water stored in glass jars for one week at 90°C. in contact with various salts and acids as Data on converted 100 gram samples of Western Electro Chemical Ore Lot No. 106 plus 200 grams indicated below.

`	I	No	ادم	r-1	Н		Ŋ			3	•		#			ľ		Ŀ	9			2		
•	75 027	Cont.	956	576	525	4.52.4	582		1163	680	513°	451.8	753.2	589	ф9	₹18₹1	η69	टभी	34.5	584 -	516	805	777	611
Ze MAAN	1	Cont.	7.04	29.8	31.9	36.3	32.8	5°62	58 2	56.1	34.5	37°4	57.5	对。8	7.44	51.03	58.9	33.5	56.1	33.6	144.1	35.44	35.5	38.85
Lty Data Cell Size	1		725	570	576	162	左9	619	629	669	159	中39	768	LTL	640I	669	724	929	68	94/	580	117	929	807
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, Crystal		Jamma	F						.4:						5		2	5	†	-+	5			5
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	7	Ems.			ig .		10	35		10	35					Я	35		10	35				-901
rta g	2011	Ton Sugar		10'-	- 35								10	35								10	35	No.
Conversion Data		wt. or salt (gms.)	29.72	29•72	29.72	75.51	55.51	35.51	71:02	71.02	71.02	37.27	57.27	57.27	43.56	43.56	43.56	87.12	87.12	87.12	28.85	28.85	28.85	Electro Lot
Salt Conve		Salt Added	- NaCI	NaCl	Macl	Ma2SOL	Ma2504	Na2SOL	Na 2501	Na2S01	Na2SOL	KC1	KCl	KCl	K2SOL	K2301	K2 SOL	K2 SOL	K2SOL	K2SOL	CaCl2	CaC12	CaC12	Western Ele

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	AA "	650 ohm		372	77.6	517		474	917	955	17	r:	01 3 3	1035	606	412	391		428	720		187						
ರ	Size	cont.	(Ers.)	30.€	†• †c	51,01	50.5	•	37.00	1,00 E	†1.††	28.2	一	十	5805	†* †*h	1,2 . 7	40 .1	61,2	5003	5.99	27.8		52.4-	37.2	36.4.	7.1	4
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	H2SOL	gmg.					 			-			121	35		+					+	CE	75		2			+0+
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sion Da	it. of	salt (ms.)	57.70	57.70	57.70	31.46	31.46	31.16	62.92	62.92	62.02	75.50	75.50	75.50	33.6	32.6	33.6	67.2	 	67.2	79.8	70.8	70.8	27.0	27.0	╁╌	7	Testorn T
Salt Conversion	Salt Added		CaCl2	CaCl2	CaCl2	MnC12	MnGl2	MnCl2	MnC12	Tnc12	PnClo	ThSO		In solt				CuC12		CuCl ₂			J. Gaga		Fed13	-		Control - Tes

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Da ta	Cell Size	\sim	(hrs.)	9.95	55.6	50.3	20.2	1.0	7.0 t	40.0	26.00		は大		results	्रविष्ठ देख	Secanse	and amps	ated out	pesnao pua	shorts.	000	600	0.10	23.4	7.0/7	52.4	55.0	20.00
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र देख	101	Sms.		C C	7 1	22								10	35		10	35					10	35		10	35	Electro	1
Conversion Data	Wt. of	salt (gms.)	81.1	81.1	81.1	1 1 1 1	00.	2.99	2.99	0.001	0.001	100.0	69.5	69.5	69.5	139.1	159.1	159.1	151.6	151.6	151.6	对•1	4.1	34.1	6.8.1	68.1	68.1	Western F	
Salt Conver	•	Salt Added	Fe013	FeCla	FeClz	7	5/thm172	Fe2(SOL)3	Fe2(SOL)3		Fe2(SOL)3	04,13	PbC12	PbC12	PbGl2	FbC12	PbC12	Pbdl2		PbSolt	Pb SOL	ZnCl2	ZhCl2	ZnCl2	ZnC12	ZnC12	ZnCl2	Cortrol - We	

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FIGURE 5. (Cont.)

													Г
Salt Conver	Conversion Data	ta ta			Crystal		Structure	ure	···	Capacity Cell	Data Size	"AA"	
	411	HC31	HOSOL,	Tene TV. Fow	Somm of	41 Th	Ro to	H. P.	25 ohm	25 ohm	56.7oh	650 ohm	Π
Salt Added	(salt)	•	Ems.	Increase		7			Lt. Ind		Cont.	Gont.	<u> </u>
$z_{ m nSOl_{ m t}}$	80.7			다	5	5	Ŧ			922	43.2		
$z_{nS0l_{\perp}}$	80.7		10	14		2	I	8		745	52.2		77
Znsolt	80.7		35	- 10			7	9		711	47.2		
CaSOL	60°98		-	*†1	7	9	E١			097	17.6		
CaSOL	60•98		10	中差	-		r-i	6		355	26.7		25
CaSOL .	60°98	and to the	35	5	-		10	E		965	21.2		
加田L) 2 ³⁰ 0 ₄	52.03		- ""	24	ત	æ	E⊣			809	1,7.0		
(1四年) 250年	20.55		10	. 3	러	6	E⊶			747	48.3		56
(虹川)250元	52.03		35	3 <u>2</u>	-	6	Н	EI,			52.6		
(时期,)2804	L0*99			2개	H	6	E→	,			1,8.7		
(NEL) 2SOL	20•99		10.	5	H	10	E		,	739	8* 11		Rī.
1082(平平)	20•99		35	z		10	Ħ		,	713	6.04		
NEI, C1	26.75			24	1	9				728	· 47•7		
инд ст	26.75	10		3 <u>2</u>		9	Ŧ	↑		713	53.5		28
MILC1	26.75	35		4		4	3	5		099	76.95		i
Lizsol	31.99			22/4	Н	6	EH	•		776	6• 44		
L1280h	31.99		្ក	3		Н	9	2		725	56.2		29
$_{ m L1_2SOl_{ m L}}$	51.99		35	2			10	EH		830	6-44		,
Lizsolt	65.98			2 <u>2</u> 2	Н	6	Ŧ				2° †19		,
11250),	65.98		ដ	3	•	EH	6	1	,	777	45.9	*	30
L12804	63.98		35	3 &	·		10		,	1168	26.0		ì
Lici .	21.5			2호		6	Ē	Н		724	54.8		
Lici	21.2	Si Ci		23		2	T	8		11,08	52.4		31
Lici	21.2	35		2			. 8	2	*****	858	70.04	•	
rol -	Western	Electro	Ľ	t No. 106	5	5	EH	; ;	912	208	38.8	644	

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Salt Conversion Data
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FIGURE 5 (Cont.)

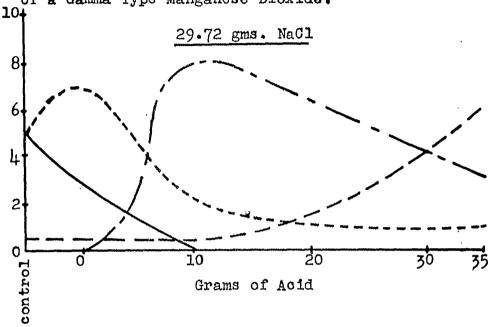
End of Data on the Introducing of Various Ions into the Crystal Structure of a Typical Garma Manganese Dioxide (Western Electro Chemical Ore Lot No. 106). Stored for One Week at 90 G. in contact with various saits and acids.

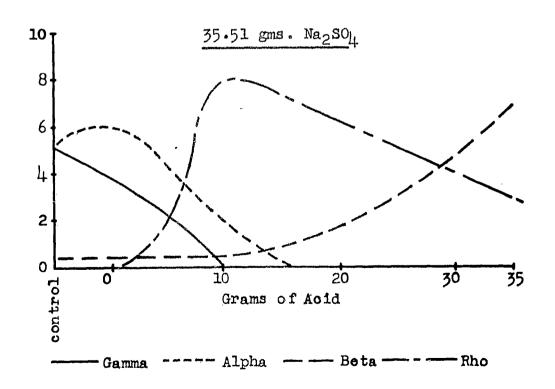
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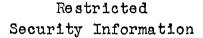
FIGURE 6

Curves Based on Crystallographic Data on the Effect of Introducing Various Ions into the Crystal Structure of a Gamma Type Manganese Dioxide.





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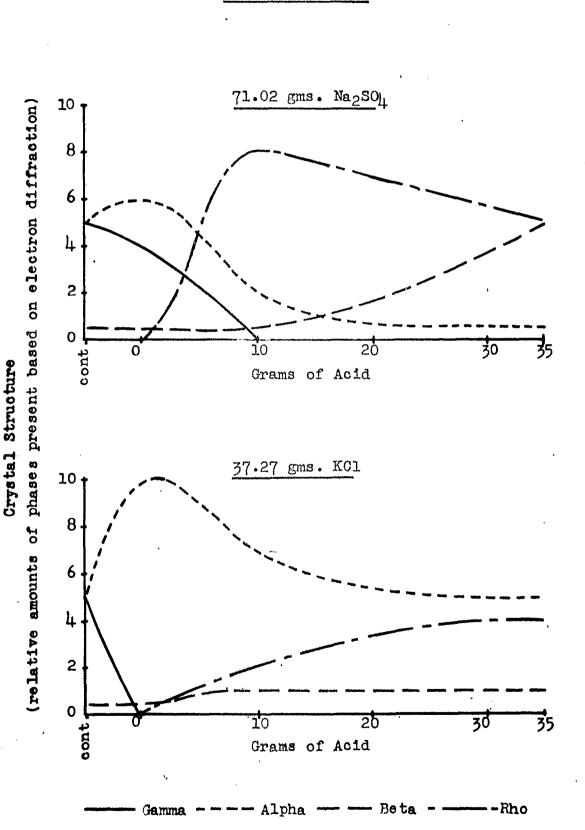
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FIGURE 6 (Cont.)



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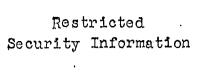


FIGURE 6.(Cont.)

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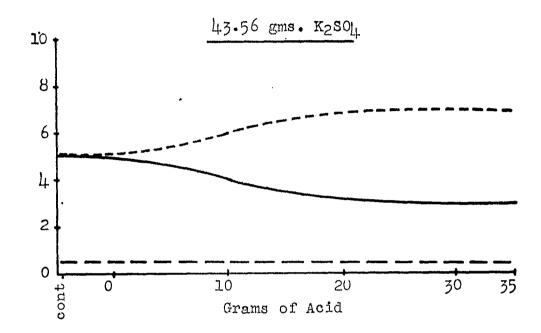
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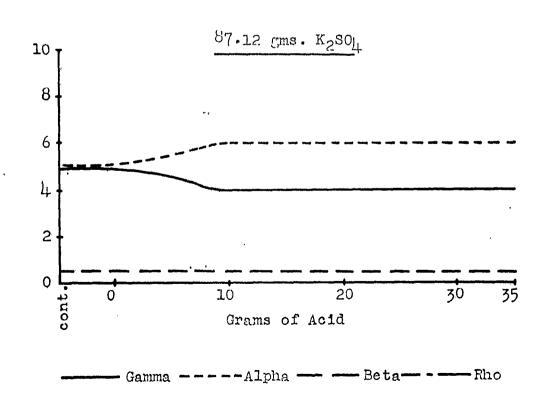
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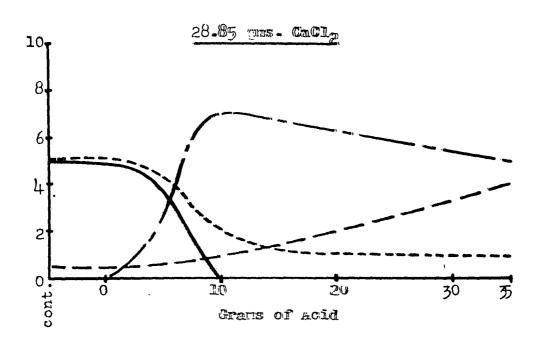
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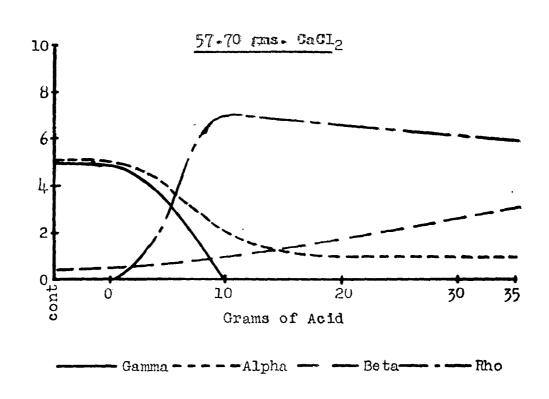
phases present based on electron diffraction)

(relative amounts of

Crystal Structure

FIGURE 6 (Cont.)





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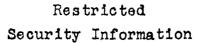


FIGURE 6 (Cont.)

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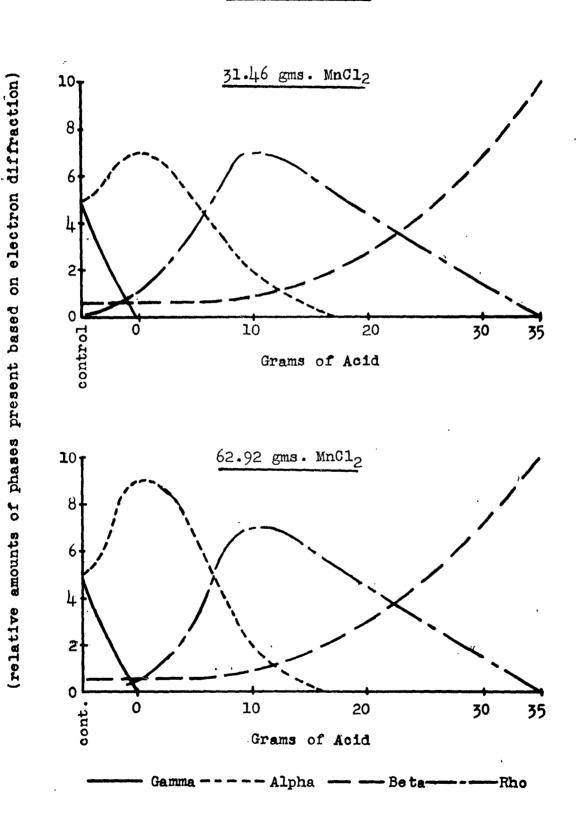
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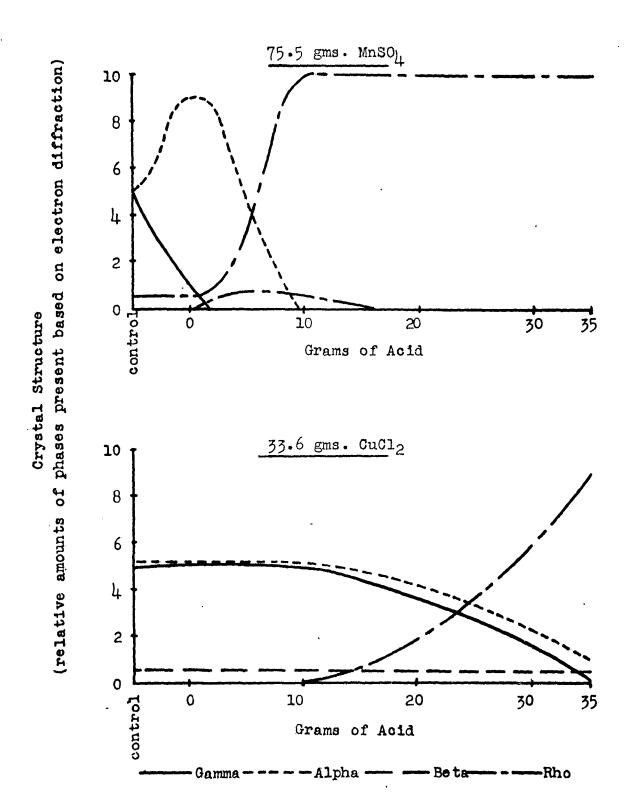
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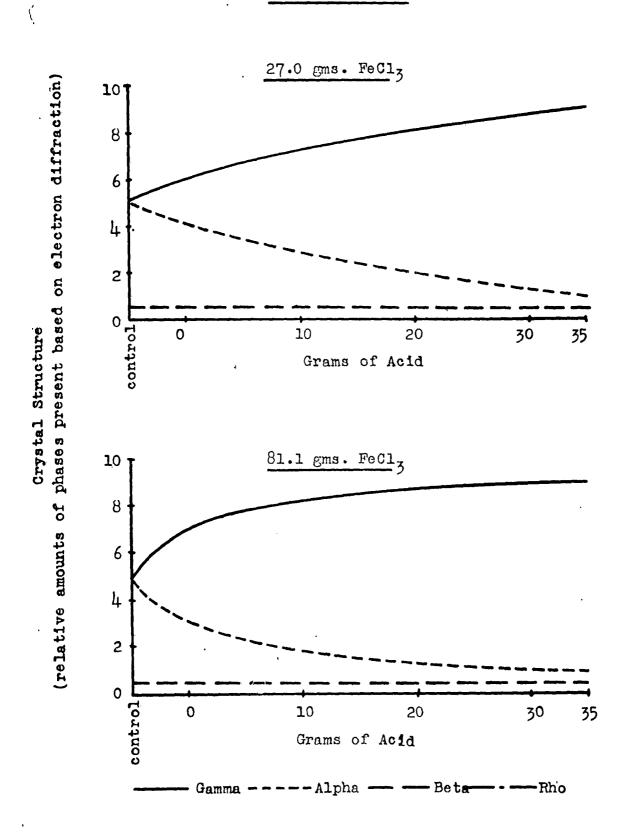
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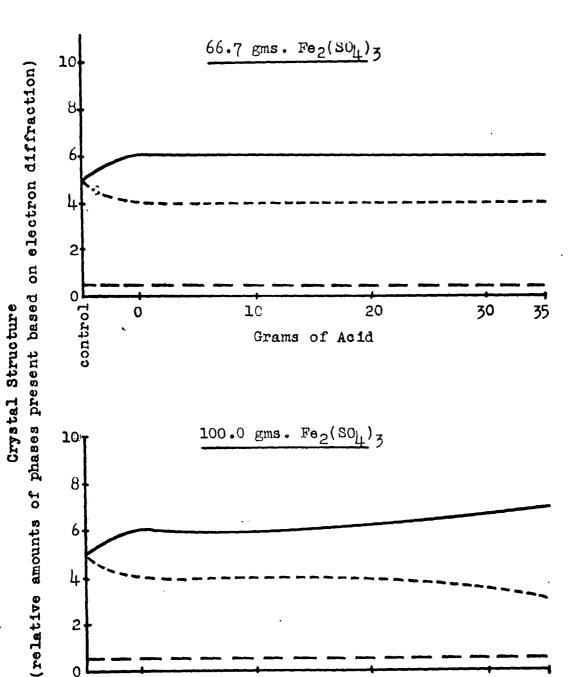
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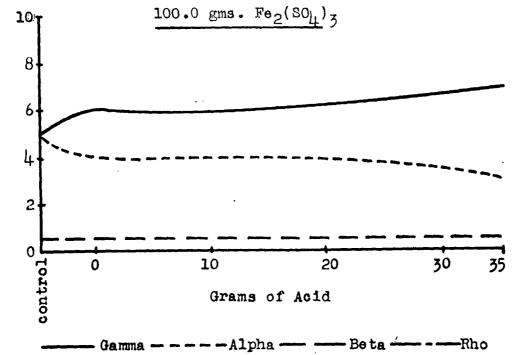
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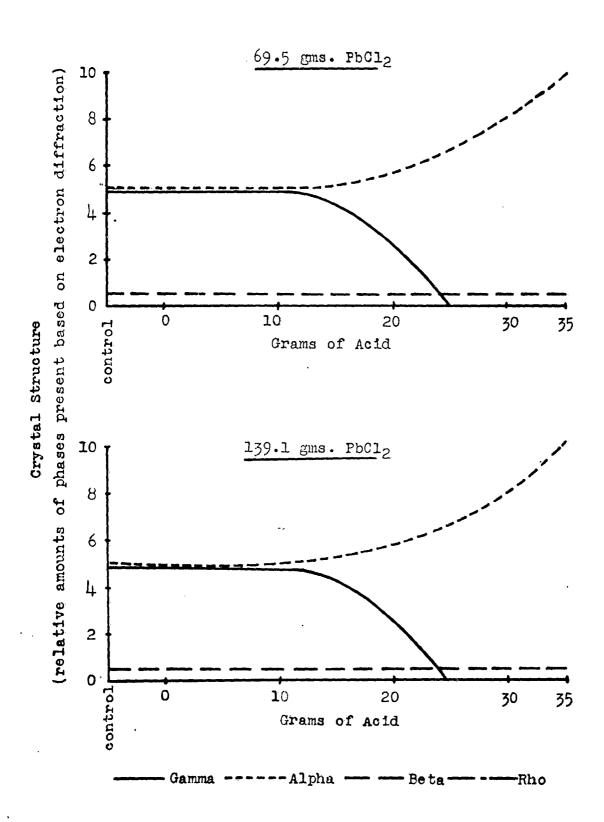
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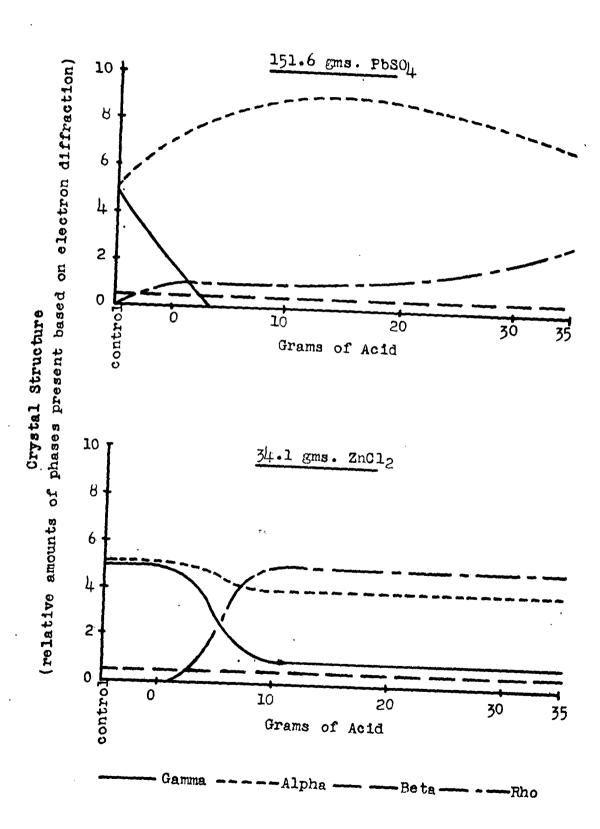




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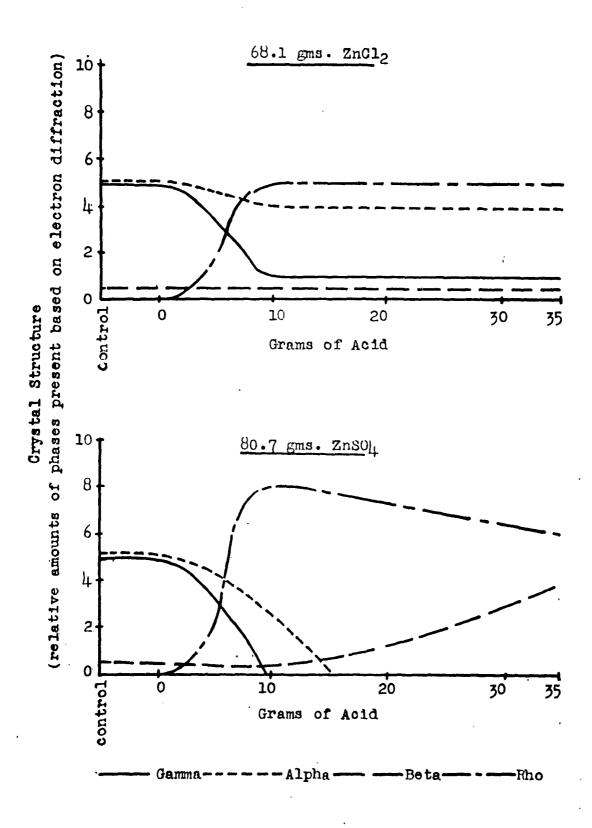


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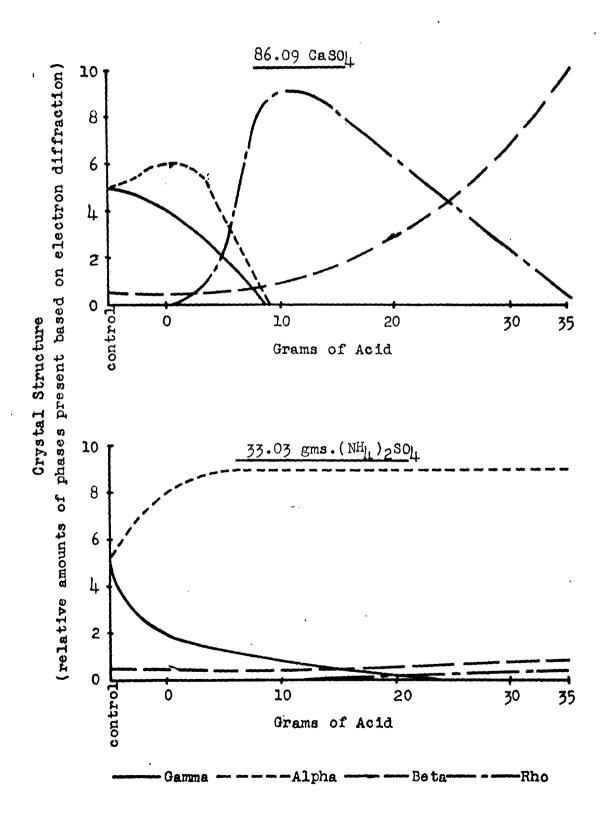
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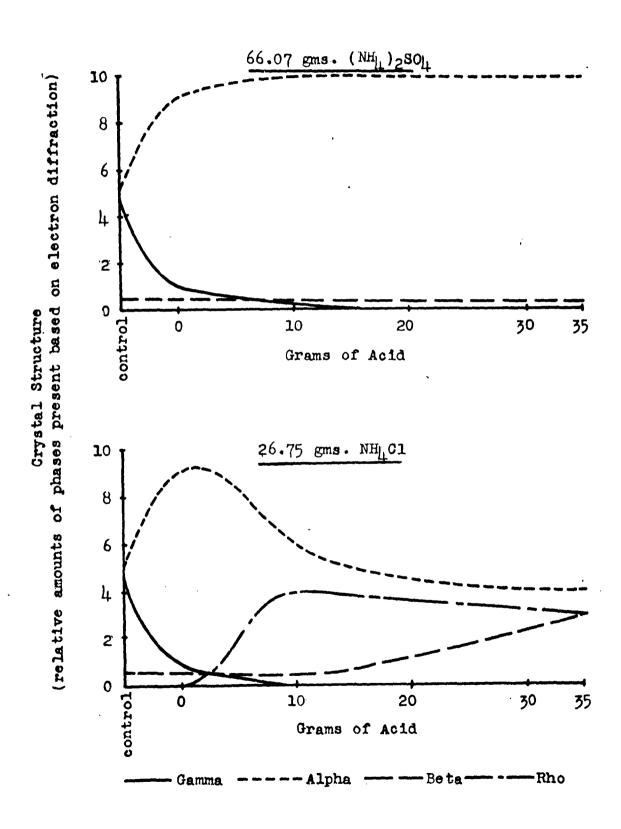


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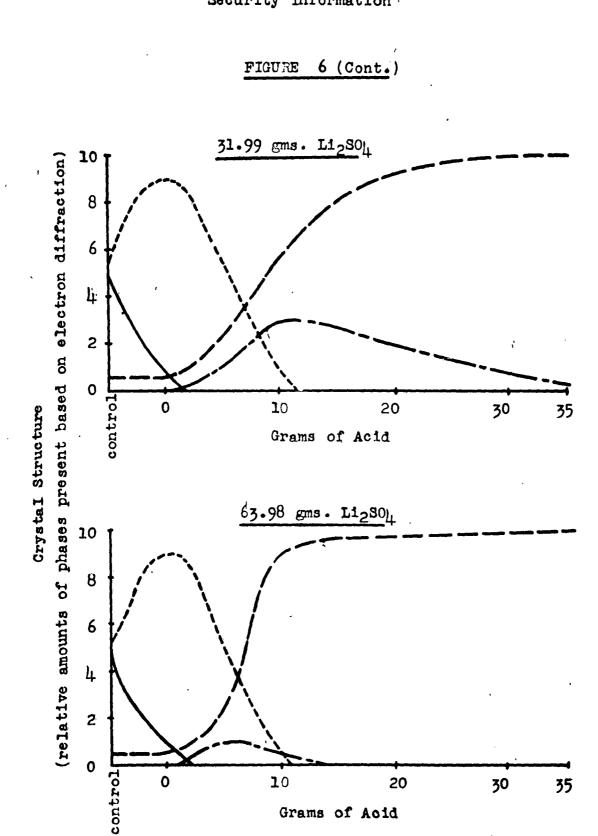


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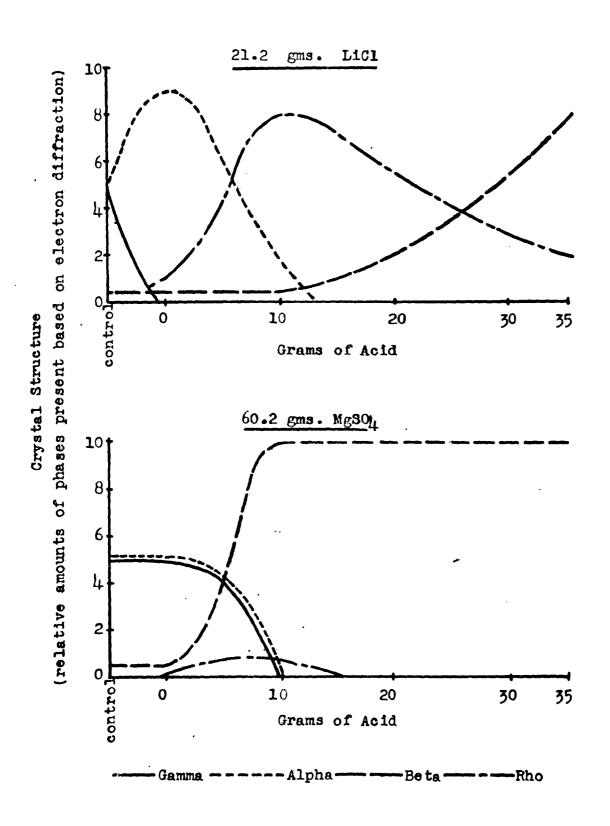
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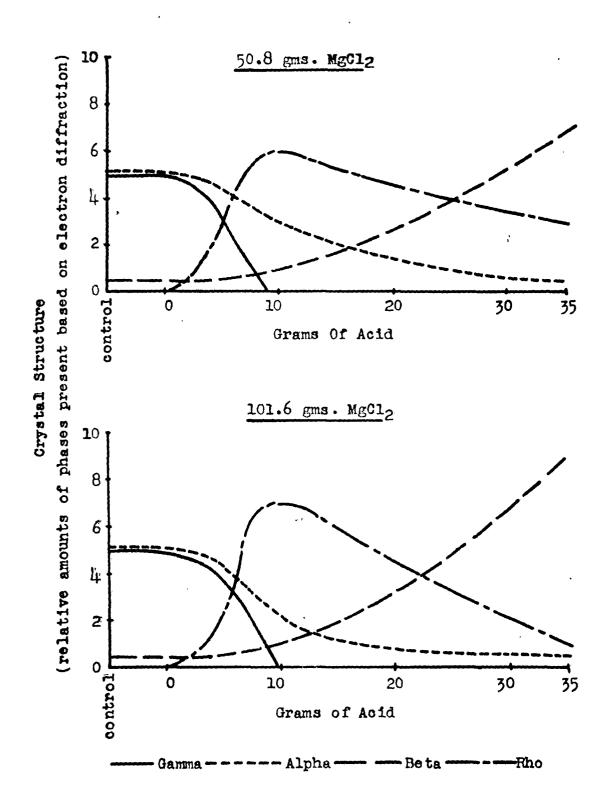


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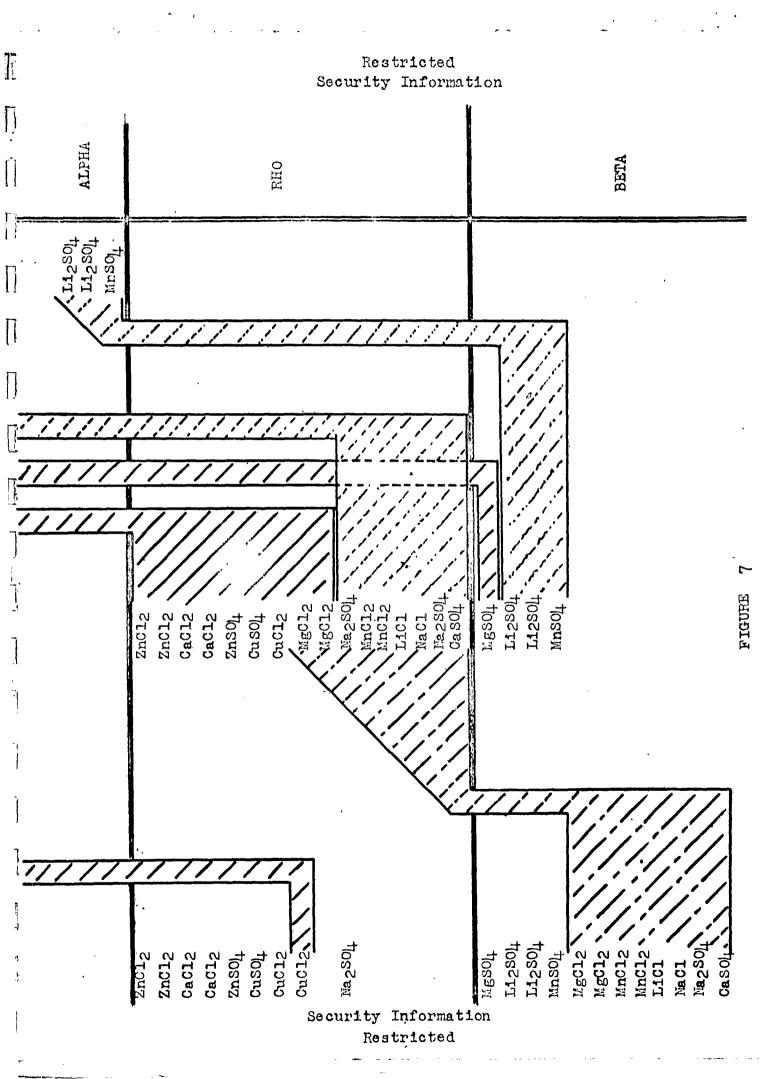
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	GAMMA	NO CHANGE (GAMMA & ALPHA)	FIGURE 7
No Acid	Fec13 Fec13 Fe2(SO ₄)3 Fe2(SO ₄)3	PbC12 PbC12 CuC12 (CuC12 (CaC12 CuC12 (CaC12 CuC12 (CaC12 (CaC12 (CuC12	Mac12 MnC12 MnC12 MnC12 MaC1 NaC1 NaC1 NaC1 NaC1 NaC1 NaC1 NaC1 N
Low Acid	FeCl3 FeCl3 Fe2(SO4)3 Fe2(SO4)3	Pb612 Pb612 Pb612	K2 SOL K2 SOL KC1 KC1 FbSOL (WH) 2 SOL
High Acid	Fec15 Fec13 Fe2(SQL)5 Fe2(SQL)3	Security Information Restricted	PbC12 FbC12 K2S04 K2S04 K2S04 KC1 KC1 FbS04 KC1 FbS04



PART III

STABILITY OF SEPARATOR MATIERALS

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Part III - Separator Materials

It is known that the voltage drop of batteries stored under tropical conditions is much more severe than the voltage drop associated with batteries stored at room temperature. Early in the contract, experiments were started to determine what effect conventional starch separators had on the delayed voltage characteristics of batteries placed on tropical storage. It was known that African ore and French Ergogene ore do not lose voltage when stored at room temperature in a sealed glass jar as a mix with Shawinigan black. This had been tested over a considerable period of time. These ores were therefore slected for preliminary investigation. Conventional D size cores were tamped from the ores. Some of the ores were stored in glass jars alone, others were cooked in starch in the jar without a zinc anode present, and others were assembled into cells in the normal manner.

The results of this study are recorded after three months in Figure #1.

The data indicate a small but definite voltage drop in the ore mixes stored by themselves. This may be attributed to either a reduction in the manganese dioxide caused by the Shawinigan black or a change in crystal structure phase resulting from the elevated temperatures.

It appears from this work that for a period of one or two months at 113°F. (depending on the ore evaluated) that both a pH change and reduction of manganese dioxide must be responsible for the voltage decrease in cells of normal construction. The increase in pH of the electrolyte can be attributed to the corrosion of the zinc. However, after this period the pH within the cell reaches a nearly constant level and reduction of the manganese dioxide by the starch becomes the more important factor.

Hypothetical curves for this analysis are shown in Figure #2.

To make certain that the starch itself was not responsible for altering the pH, a blank was run in which battery electrolyte and starch were cooked together at a given pH and then stored for a period of two months at 113°F. It was found upon re-measuring the pH that no appreciable change had taken place. Actually the pH had dropped from an initial value of 3.6 to a value of 3.2. This indicates that the voltage drop of the manganese dioxide mixes stored in contact with the starch alone was due entirely to the reduction of the manganese dioxide by the starch. Chemical action accelerated by high temperature causes serious voltage drops at temperatures of 130°F. and 160°F. presenting a serious problem to high storage conditions.

In order to prove that a conventional separator will substantially reduce the voltage of manganese dioxide, the individual components of a paste separator (corn starch, potato starch, flour, and electrolyte), were tested. Several different types of manganese dioxide (African ore, Baker's Analytical (pyrolusite), Burgess Chem ore, Burgess Electro ore and cryptomelane ore made by leaching light hydrate in 20% zinc chloride solution) were used in these

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tests to determine if the structural form of manganese dioxide contributed in any manner to the amount of voltage reduction.

The effect of separator materials was tested in the following manner. Using a conventional electrolyte as a wetter, cores of AA size were made from 100% mixes (7/1 - ore/black) of the five manganese dioxides listed above. The separator materials being tested, namely, corn starch, potato starch, and flour, were then made into pastes using the same electrolyte that was used as a wetter when making the manganese dioxides into mixes. The AA cores were placed in these separator pastes in glass jars and the pastes gelled by heating. The glass jars were then sealed and placed in storage at 113°F. Glass jars were used in order to eliminate the problem of pH change caused by the presence of a zinc anode. The paste gel surrounding the AA cores was about five times the thickness found in a conventional cell. This thickness was used in order that sufficient separator would be present to give substantial evidence of reduction in the voltage of manganese dioxide. Control samples for each type of manganese dioxide were also stored at 113°F. The control consisted of samples of the individual mixes, stored in glass jars, i.e. with only electrolyte present.

The voltage of the different samples was measured initially and after three days at 70°F, and after two weeks and one month at 113°F. The table and graphs showing one month reduction in voltage of these samples are given in Figure #3, While Figure #4 in the series shows voltage reduction at the end of six months with African ore. Data accumulated over a twelve month period for these samples are given in Figure #5 along with the graphic chart of the effect of corn starch samples on these ores.

These results show that a considerable drop in voltage is caused by the materials tested. Greatest reduction in voltage is evidenced in the case of the flour separator while potato starch appears to be the best. The graphs show that the reduction in voltage is very rapid under the conditions present over the first part of the storage period but has a tendency to level out during the latter part of the storage period.

The data indicate that the greater the degree of activation of the mangamese dioxide the greater will be the voltage reduction which takes place.
For example, African ore stored in contact with corn starch underwent a voltage
decrease of 0.22 volts, while Burgess Electro ore underwent a decrease of
0.38 volts under the same conditions. The initial voltage of the Burgess
Electro ore sample was approximately 0.10 volts above the initial voltage of
the African ore sample, while after twelve months storage at 113°F it was
approximately 0.05 volts below the voltage of the African ore sample.

Starch chemistry indicates that once oxidation and breakdown of the separator material begins, it continues with greater ease. A higher voltage manganese dioxide should, therefore, give rise to an increased initial oxidation and breakdown.

The voltage readings of the cores in contact with separator materials

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were measured in the following manner. One leg of a modified salt bridge (see Figure I) was filled with the same electrolyte used in the samples, and was placed in contact with the separator materials in the glass jars. The second leg of this salt bridge (containing saturated KCl solution) was in contact with a calomel half cell. A lead wire from the catomel cell to the negative emf terminal of a potentiometer and a lead wire from the carbon rod of the AA core in contact with the separator material to the positive emf terminal of the potentiometer completed the circuit. The voltage of the manganese dioxide minus that of the calomel cell was read directly on the potentiometer. Thus by adding the voltage of the calomel cell (0.242V) to the reading obtained from the potentiometer, the voltage of the manganese dioxide was obtained. Since the potetiometer used was a null instrument, no drain was put on the sample. Thus readings can be repeated as often as desired with no destructive effects to the samples.

The voltage of the control samples was measured in a slightly different manner since they were stored as mixes rather than as tamped cores. The procedure used for determining the voltage of these control samples was as follows. A hole, approximately 3/32% in diameter and 1/8% deep, was drilled in a carbon rod and a small quantity of mix solidly tamped into this cavity. This rod had a brass cap on the other end to insure good contact. This assembly was then pressed tightly against an absorbent piece of Crocker-Burbank tan saturating paper, the paper first having been saturated with the electrolyte used to wet the mixes initially. The leg of the modified salt bridge containing electrolyte was also pressed against the saturated paper a slight distance away from the carbon rod assembly. The saturated paper made a conductive path between them. The measurement of the voltage of the sample was then carried out as described in the preceding paragraph.

A study was made to determine if the starch had to be in contact with the manganese dioxide. The data are shown in Figure 6. It can be seen that when the manganese dioxide was separated from the starch by an electrolyte barrier a smaller decrease in voltage resulted than when the starch was in direct contact. However, this difference is quite small when compared to the overall reduction in voltage which occurs. The study definitely shows that the starch does not have to be in contact with the manganese dioxide in order to seriously reduce its voltage and that the major proportion of the voltage reduction is caused by some soluble components already present in the starch or by some component formed on the acid action of the electrolyte on the starch.

A study was made to determine the effect of paste thickness on the voltage of manganese dioxide. The data are shown in Figure 7. It can be seen that the amount of reduction in voltage varies directly with the paste thickness. This is probably due to a large percentage of soluble material being present due to the extra starch.

Figure 8 is a study of the effect of varying the electrolyte pH on the oxidation of flour (manganese dioxide voltage reduction) when stored as a

SECURITY INFORMATION RESTRICTED paste in contact with manganese dioxide at 113°F. Control samples were made by wrapping AA size cores with dynel cloth and storing them in an electrolyte whose pH had been adjusted by the addition of ammonium chloride. The test samples were made by storing AA size cores in a flour paste made with the adjusted pH electrolyte. Electrolytes from a pH of 3.8 to a pH of 7.9 were studied.

As would be expected, the initial voltage of the manganese dioxide decreases as the pH rises. After six months of storage at $113^{\circ}F_{\circ}$, the manganese dioxide stored in contact with the flours eparator shows the greatest voltage reduction at the lowest pH_{\circ} , and the amount of reduction decreases as the pH_{\circ} is increased, as illustrated by the accompanying chart. The formation of diammine occurs at about $pH_{\circ} = 5.2$ with the electrolyte used.

Above the pH of 5.2 the voltage reduction is essentially constant. The varying pH of the electrolyte does not appear to significantly affect the voltage reduction of the control samples.

This study along with the study of the effect of electrolyte pH on the voltage of minc indicates the cells utilizing a pH adjusted electrolyte may have benefits.

The fact that voltage reduction is lower at a less acid pH tends to prove that at least some of the voltage reduction is due to components formed by the acid action of the electrolyte on the starch molecule.

The search for a suitable separator included a wide variety of possible materials which were tested on storage at 113°F. and 160°F. in a manner previously described to determine the extent to which they will reduce the manganese dioxide.

These materials included various starches, gums, proteins and glutens. The results of these tests showing the most promising materials are tabulated in Figure #9 for 113°F., and in Figure #10 for 160°F.

The proteins and glutens were tested in much the same manner as the starch materials. Since the proteins and glutens are for the most part only slightly soluble in electrolyte, a saturated solution (containing about 1% of the material) was made up. Double AA cores were tamped from an African ore mix (7/1 ore/black) and wrapped with Dynel cloth, which is a non-woven fabric having no reducing affect on manganese dioxide. The wrapped cores were then placed in sample bottles containing the various saturated solutions and placed on storage. Results indicate that the proteins and glutens (at least in the amounts used) do not reduce the voltage of manganese dioxide as greatly as do corn or potato starch. In addition, both proteins and glutens show good inhibiting properties and it is quite possible that they may be incorporated into a separator material.

Those of a protein nature do not form gels and therefore it would be

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necessary to use them with some other separator material, probably in the capacity of an inhibitor.

On the basis of this evaluation work a definite correlation can be set up in regard to the materials tested. As a result the following classifications can be used in regard to separator materials.

- (a) The first classification is headed by conventional corn and potato starches and includes those materials which cause a voltage decrease of approximately 0.2 to 0.25 volts over a period of one year. This class includes almost all of the various starch type materials.
- (b) The second classification includes the various flours and shows a voltage decrease of approximately 0.25 to 0.30 volts.
- (c) The third classification includes materials which cause comparatively little voltage reduction during the storage period. This classification includes such materials as proteins, glutens and most of the gum type materials. These materials cause a voltage reduction of approximately 0.10 to 0.15 volts during the storage period.

The above classifications are based on the results of twelve months tests conducted on separator materials in contact with African ore. The same classes exist when other ores are used but will vary in the degree of voltage reduction.

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Conclusions

- 1. Starch and flour materials substantially reduce manganese dioxide.
- 2. Four reduces manganese dioxide more than starch.
- 3. The voltage reduction of a manganese dioxide when stored in contact with a conventional separator material will be greater as the degree of activation of the ore is increased.
- 4. Increasing the amount of starch in contact with manganese dioxide increases the voltage reduction of the ore.
- 5. If the pH of the electrolyte used in a starch paste is increased, the percentage of voltage drop in manganese dioxide is decreased until the pH at which diammine forms is reached. After this point the reduction is essentially constant with a pH rise.
- 6. Most starch materials will not retain their gel structure when stored as paste mixture in contact with manganese dioxide for extended periods at 160°F.
- 7. The reduction in voltage of manganese dioxide when stored in contact with starch is due to some soluble product initially present in the starch and/or some product formed by the acid action of the electrolyte on the starch.
- 8. Reduction in voltage of manganese dioxide due to starch oxidation is rapid during the early portions of storage (one month at 113°F, and two weeks at 160°F). The voltage tends to level out with little change in the later stages of storage.

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TABLES AND CHARTS

PART III

STABILITY OF SEPARATOR MATERIALS

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FIGURE 1

Manganese Dioxide Voltage De	crease	Duo T	o Oxi	dation	n Of Sta	irch S	epare	tor
•					rage of	U	Read	lings
		All r	oadin	gs ma	de at 70) F.		
Cell Construction	French	Ergo	gene	Ore	A1	frican	ore	
					Initia			
	@ 70°F.	113 F	113 F	113 F	@ 70°F	113 F	113 F	1137
Mix tamped into "D" size				ļ <u> </u>				
slug with no pencil. Slug								
stored in sealed glass	1.82	1.80	1.78	1.70	1.67	1.66	1.65	1.60
jar without separator or	v.	v.	٧.	v.	٧.	₹.	v •	٧.
anode.					ļ			
Mix tamped into "D" size						•		
slug with no pencil. Slug								
stored in excess flour-	1.82	1.63	1.57	1.45	1.67	1.58	1.58	1.50
starch paste separator	[
with no anode present.					ļ			
Mix tamped into convent-						<u> </u>		
ional "D" size bobbin.	_							
Stored in excess flour-	1.82	1.63	1.56	1.42	1.67	1.58	1.58	1.51
starch paste separator]						-	
with no anode present.								
Mix tamped into convent-						'		-
ional "D" size bobbin.								
Bobbin stored with no	1.82	1.79	1.74	1.65	1.67	1.65	1.64	1.58
anode or separator present	ļ			ļ.	I			
						<u> </u>		
Conventional "D" cell stor						·		
ed for desired period.					1			\
Bobbin rémoved from can	1.82	1.62	1.58	1.57	1.67	1.58	1.56	1.54
at end of storage and] .	Ì			
made into fresh cell.								
Conventional "D" size bob-]			
bin stored in excess flour	h _ /							
-starch separator with no	1.82	1.65	1.57	1.48	1.67	1.61	1.60	1.55
anode. At end of storage		<u>'</u>			1			
time made into fresh cell.								

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FIGURE 1 (Cont.)

Manganese Dioxide Voltage Decrease Due To Oxidation Of Starch Separator

	Volt				rage of made at	_		lings
Cell Construction	French	Ergo	gene	Ore	Αi	ricar	ı Ore	
					Initial @ 70°F.			
Conventional "D" size bob- bin stored with no separ-								
ator or anode. Bobbin re-	1.82	1.80	1.77	1.71	1.67	1.68	1.68	1.63
moved at end of storage and made into fresh cell.	v•	v.	▼•	v.	٧.	▼.	v.	۷.
Conventional "D" size, bobbin stored in the presence of glass wool and ZnCl2 TNH ₁ Cl solution.	. 0.	1.75 pH of sol.	1.73 pH of sol.					
migor boracions			3.33					
Conventional "D" size bob- bin stored in the presence	}	1.71	1.59	<u> </u> - 				
of glass wool, ZnCl2- NH4Cl solution and a zine mesh anode.	1.82	pH of sol. 4.2	pH of sol.					

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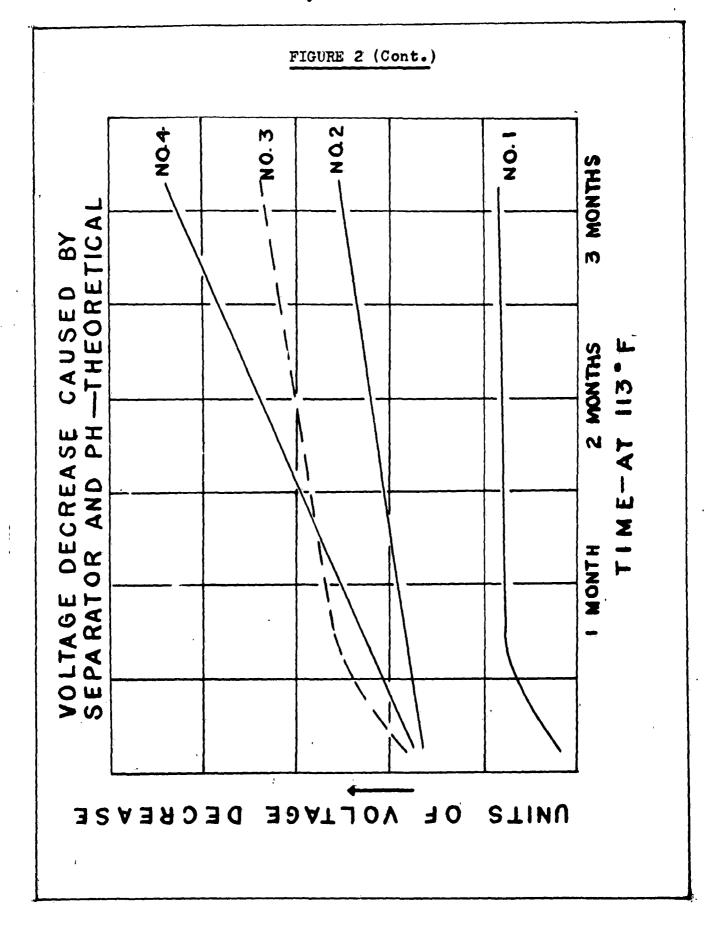
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FIGURE 2

Theoretical Curves of Voltage Decrease Due to the Effect of Starch Oxidation and the Change of pH.

- Curve No. 1 Voltage decrease due to the change of pH (corrosion).
- Curve No. 2 Voltage decrease due to exidation of starch only normal starch separator thickness (no pH factor).
- Curve No. 3 Voltage decrease due to change in pH plus voltage decrease due to oxidation of starch (normal cell).
- Curve No. 4 Voltage decrease due to oxidation of starch only excess starch (no pH factor).

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FIGURE 3

Study of Voltage Decrease of Depolarizer Mixes Due to the Oxidation and
Breakdown of Separator Materials

	<u>, </u>			r Material:	<i>-</i>	
Depolarizer	Separator	· 	Voltago R			Voltage
Mix	Materials	Initial	3 days © 70 ⁰ F.	2 weeks @ 113 ⁰ F.	1 month @ 113 ⁰ F.	Drop Afte: 1 month
,	Control-only Electrolyte	0.920 v.	group and then see path done gar.	,	0.914 v.	0.006 v.
100%	Corn starch	0.920 v.	U.863 v.	0.812 v.	0.800 v.	0.120 v.
African Ore	Potato Starch	0.920 v.	0.88ft A.	0.821 v.	0.805 v.	0.115 v.
•	Flour	0.920 v.	0.788 v.	0.724 v.	0.724 v.	0.196 v.
	Control-only Electrolyte	0.895 v.			0.894 v.	0.001 v.
100%	Corn starch	0.895 v.	0.812 y.	0.736 v.	0.716 v.	0.179 v.
Bakers · Pyroluscite	Potato Starch	0.895 v.	0.836 v.	0.776 v.	0.740 v.	0.155 v.
	Flour	0.895 v.	0.775 v.	0.695 v.	0.673 v.	.0.222 v.
	Control-only Electrolyte	0.995 v.	450 De De De De De		0.980 v.	0.015 v.
100%	Corn starch	0.995 v.	0.902 v.	0.789 v.	0.744 v.	0.249 v.
Burgess Chem. Ore	Potato starch	0.995 v.	0.922 v.	0.80l ₊ v.	0.750 v.	0.245 v.
	Flour	.0 • 995 v •	0.813 v.	0.686 v.	0.665 v.	0.330 v.
	Control-only Electrolyte	1.025 v.			1.025 v.	0.000 v.
100%	Corn starch	1.025 v.	0.957 v.	0.816 v.	0.756. v.	0.269 v.
Burgess Electro Ore	Potato starch	1.025 y.	0.982 v.	0.812 v.	0.762 v.	0.263 v.
	Flour	1.025 v.	0.855 v.	0.754 v.	0.684 v.	0.341 v.
100%	Control-only Electrolyte	0.962 v.	top use two year top got	\$10 to. No. (*) 40 gp.	0.931 v.	0.031 v.
Cryptome lane	Corn starch	0.962 v.	0.855 v.	0.738 v.	0.705 v.	0.257 v.
(Lt. Hydrate leached in	Potato starch	0.962 v.	0.881 v.	0.761 v.	0.670 v.	0.292 v
20, ZnCl2 sol	k'Lour	0.962 v.	0.781 v.	0.686 v.	0.670 v.	0.292 v

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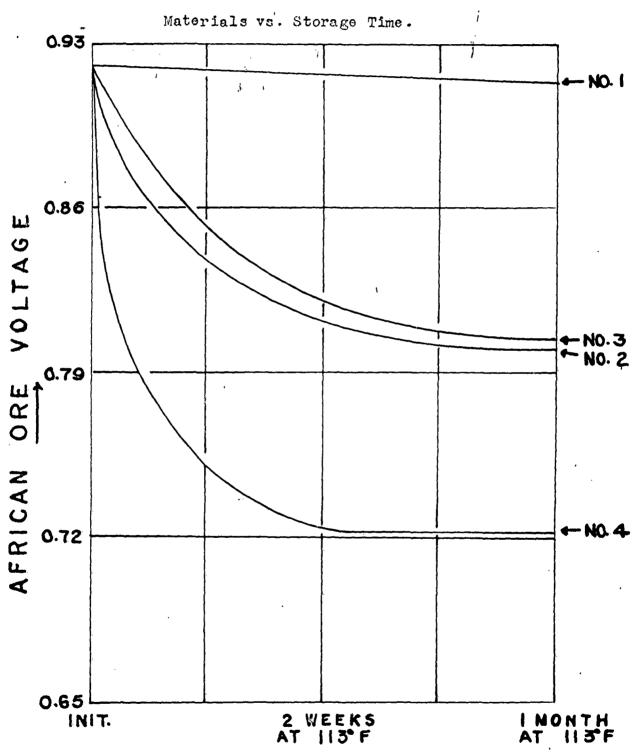
The voltage of Mangarese Dioxides in Contact with Separator Materials versus Storage Time at 113°F.

- Stored in contact with electrolyte only.
- Curve No. 2 Manganese dioxide mix (7/1-ore/black) stored in contact with a corn starch-electrolyte gel.
- Curve No. 3 Manganese dioxide mix (7/1-ore/black) stored in contact with a potato starch-electrolyte gel.
- Curve No. 4 Manganese dioxide mix (7/1-ore/black) stored in contact with a flour- electrolyte gel.

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Voltage of African Ore in Contact with Separator

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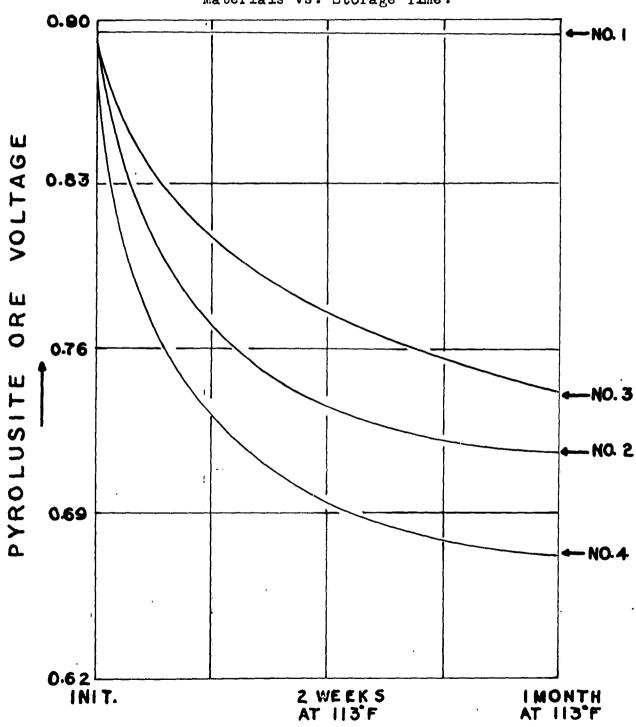


STORAGE TIME

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Voltage of Pyrolusite Ore in Contact with Separator

Materials vs. Storage Time.

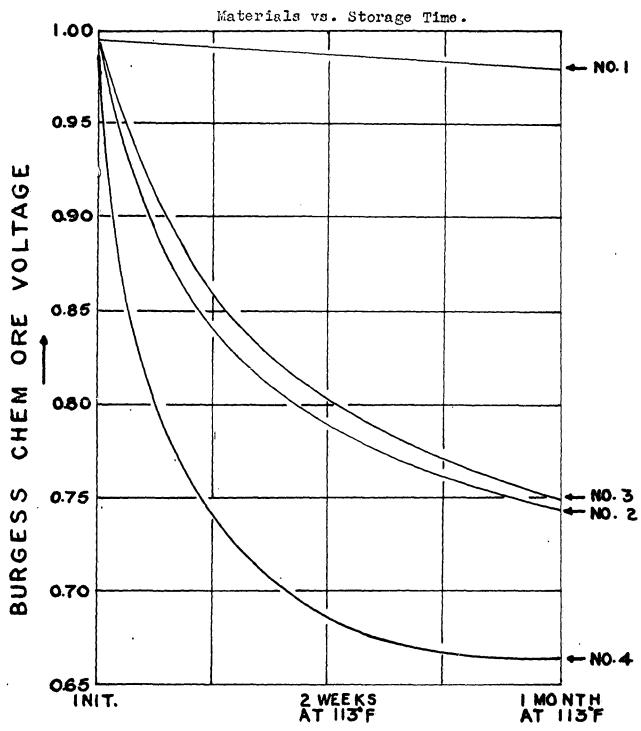


STORAGE TIME

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Voltage Of Burgess Chem Ore in Contact with Separator



STORAGE TIME

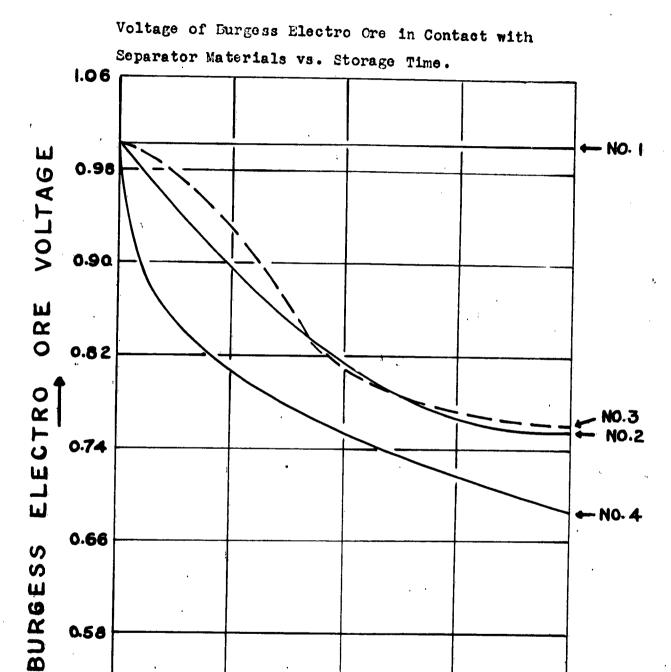
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Retake

I MONTH

AT 113°F

FIGURE 3 (Cont.)



STORAGE TIME

2 WEEKS

AT 113° F

0.58

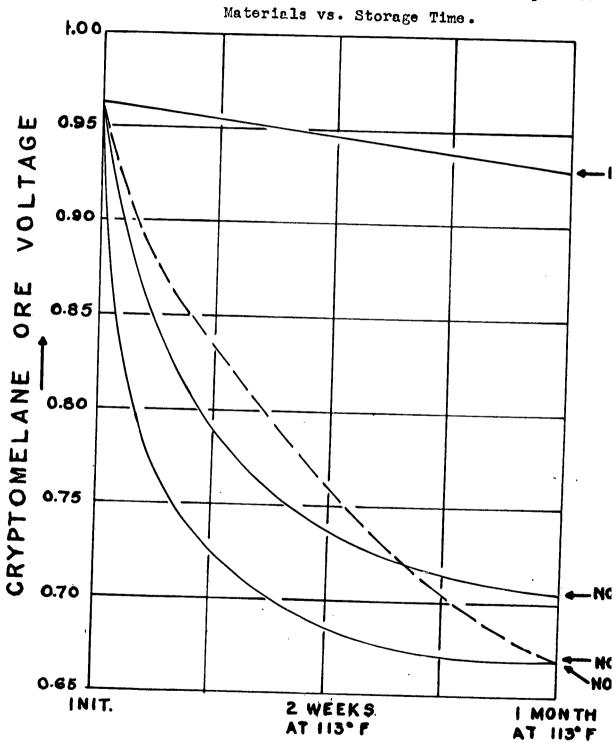
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Voltage of Cryptomelane Ore in Contact with Separator
Materials vs. Storage Time.



STORAGE TIME

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Voltage of African Ore in Contact with Separator Materials vs. Storage Time After Six Months Storage

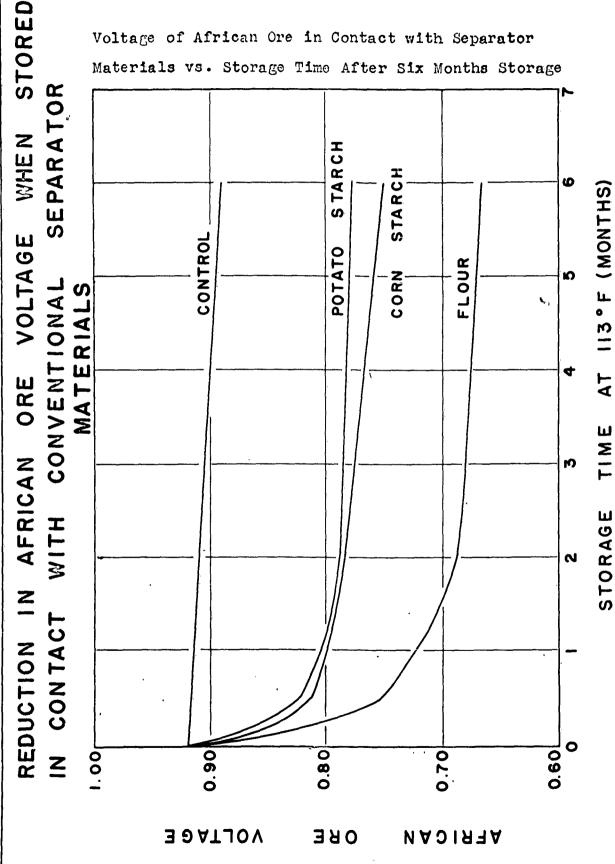
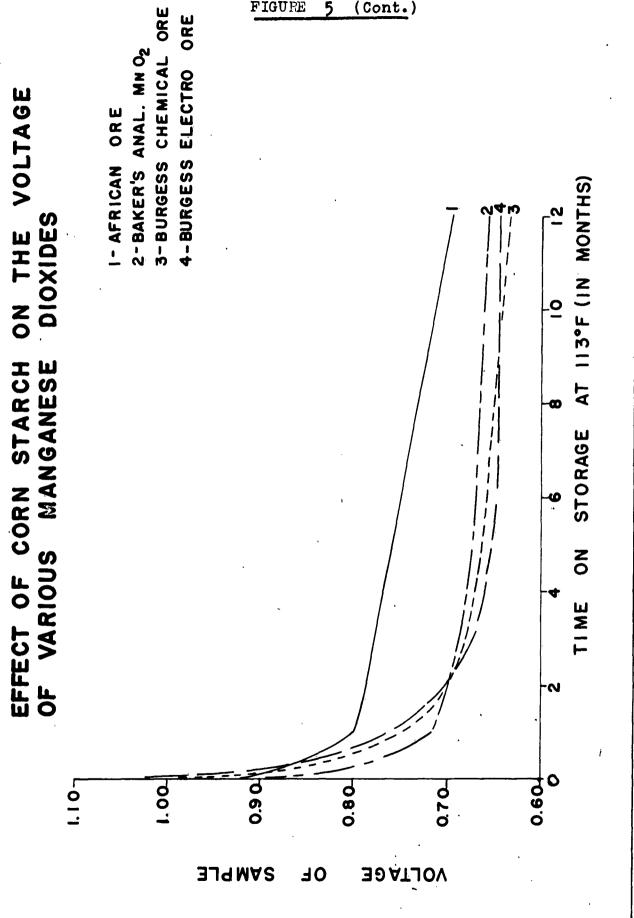


FIGURE 5

Comparison Of Voltage Reduction Caused By Common Separator Materials When Stored In Contact With Various Types Of Manganese Dioxide

}			2000					
Lix For	Lix Formulation		ore/black -	ore/black - 7/1 ("AA")				
Electro	Electrolyte Formulation	24.	0% NH1,C1; 2	24.0% MH1G1 ; 22.5% ZnCl2 ; 53.5% H2O	53.5% H20			
Storage	Storage Conditions	Sea	led glass ja	Sealed glass jars - no zinc present	present			
Separato	Separator Thickness	0.h	0.438 inch					
Camile	***************************************	3.0	Voltage of	Voltage of sample after storage at 113° F.	r storage at	113º F.		- 1
No.	Material	Tested	Initial	1 month at	3 months at 6 months at 12 mos. 113°F 113°F	6 months at	12 mos. at	Voltage Reduction
νī	Corn Starch	African	0.920 v.	0.300 v.	0.780 v.	0.750 v.	0.702 v.	0.218 v.
2.4	Potato Starch	Cre	0.920 v.	0.805 .	0.785 v.	0.770 v.	0.722 v.	0.198 v.
33	Flour		0.920 v.	0.724 v.	0.684 v.	0,666 v.	0,66μ ν.	0.256 v.
1B	Corn Starch	Baker's	0.895 v.	0.716 v.	0.688 v.	0.668 v.	0.654 v.	0.241 v.
E Z	Potato Starch	Anal.	0.895 v.	0.740 v.	° γ 769°0	0.65h v.	0.658 v.	0.237 v.
恩	Flow	Mr02	0.895 v.	0.672 v.	° 0999°0	0.650 v.	.0.645 v.	0.250 v.
10	Corn Starch	Burgess	•a ≤36•0	0.744 v.	0.681 v.	0.659 v.	0.535 v.	0.360 v.
3C	Potato Starch	Chemical	0.995 v.	0.750 v.	0.682 v.	0.654 v.	0.536 v.	0.359 v.
30	Flour	O re	0.995 v.	0.665 v.	0.643 v.	0.638 v.	0.622 v.	0.373 v.
£	Corn Starch	Burgess	1.025 v.	0.755 v.	0.670 v.	0.647 v.	0.645 v.	0.380 v.
20	Potato Starch	Electro	1.025 v.	0.762 v.	0.677 v.	0.647 v.	0.646 v.	0.379 v.
30	Flour	Ore	1.025 v.	°4 €89°0	0.653 v.	0.644 v.	0.625 v.	0.400 v.



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5 (Cont.) ESE DIOXIDES STARCH ORE MN 02 ORE MANGANESE ELECTRO CHEM. ANAL. CORN TIME ORE TIME (MONTHS) BURGESS BURGESS BAKER'S AFRICAN WITH STORAGE E OF VARIOUS CONTACT WIT **S** \ STORAGE E DECREASE STORED IN (VOLTAGE 0.50⁻ 0.30 0.40 0.20 0.10 VOLTAGE O F STINN DECKEASE Security Information

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Study To Determine If Starch Must Be In Contact With Manganese Dioxide In Order to Reduce the Voltage Of The Manganese Dioxide

Sample		Voltage of s	ample after	storage
No.	Method of Testing	Initial	l mo. at 113°F.	Voltage Reduction
0	Control - Direct contact between starch and manganese dioxide (African ore)	0.920 v.	0.765 v.	0.155 v.
1	Starch separated from	0.920 v.	0.784 v.	0.136 v.
2	manganese dioxide by Dynel cloth and an	0.920 v.	0.783 v.	0.137 v.
3	electrolyte barrier	0.920 v.	0.807 v.	0.113 v.
4	Manganese dioxide in contact with electrolyte only.	0.920 v.	0.914 v.	0.006 v.

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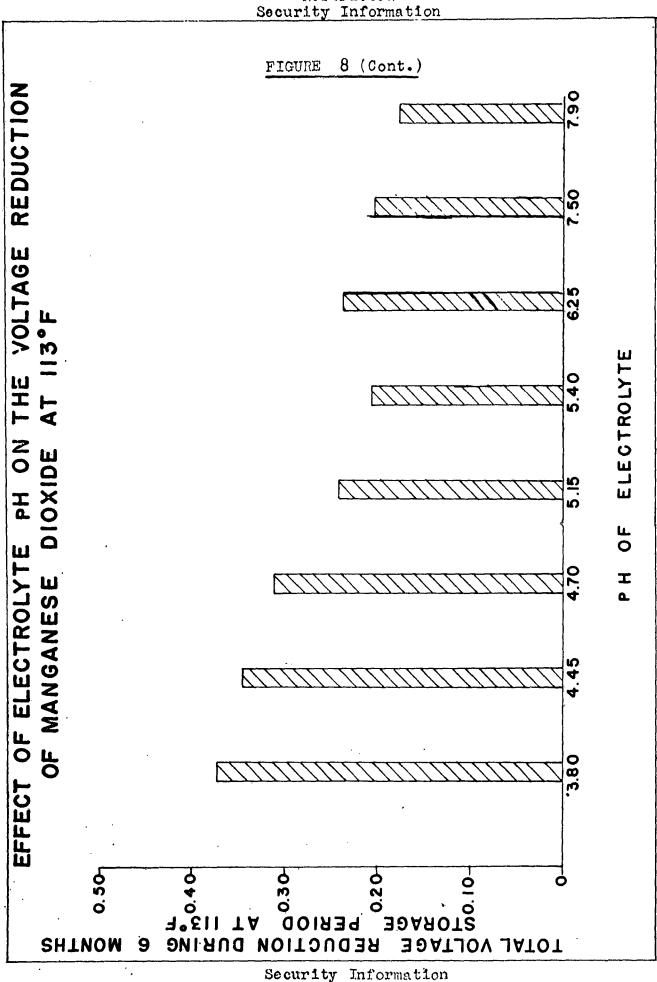
Study Of The Effect Of Paste Of Varying Thicknesses On The Voltage Of Manganese Dioxide

Ca		D4-	0	Voltage of	sample	Amount of
Sample No.	Core D <u>i</u> ameter	Paste Thickness	Ore Tested	Initially	1 month at 113°F	reduction in voltage
1	0.375 in.	0.093 in.	Africar Ore	0.920 v.	0.814 v.	0.106 v.
2	0.375 in.	0.156 in.	11	0.920 v.	0.794 v.	0.126 v.
3	0.375 in.	0.188 in.	11	0.920 v.	0.788 v.	0.132 v.
4	0.375 in.	0.281 in.	и .	0.920 v.	0.780 v.	0.140 v.
5	0.375 in.	0.438 in.	11	0.920 v.	0.772 v.	0.148 v.
6	0.375 in.	0.531 in.	п	0.920 v.	0.766 v.	0.155 v.

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т. т	u L	11	т.	•

Effect Of Electrolyte pH On The Oxidation Of Flour Paste When Stored As A Paste In Contact With Manganese Dioxide At 113°F.

Electrolyte Formulation		24.0% NH4Cl; 22.5% ZnCl2; 53.5% H2O (Composition by weight) NH4OH added to bring about pH rise				
Ore Te	sted	Burg	ess Electro ore mix	as "AA" c	ores	
Sample No.	pH of Electrolyte		Separator Material	Voltage of Initial		Voltage Reduction
6 0	3.80	,	Flour Paste	1.024 v.	0.652 v.	0.372 v.
60A	3.80		Control - Electro- lyte only	1.062 v.	1.004 v.	0.058 v.
61	4.45		Flour Paste	0.988 v.	0.645 v.	0.343 v.
61A	4.45		Control	1.038 v.	1.002 v.	0.006 v.
62	4.70		Flour Paste	0.950 v.	0.640 v.	0.310 v.
62A	4.70		Control	0.996 v.	0.996 v.	0.000 v.
63	5.15		Flour Paste	0.875 v.	0.635 v.	0.240 v.
63A	5.15		Control	0.993 v.	0.961 v.	0.032 v.
64	5.40		Flour Paste	0.831 v.	0.628 v.	0.203 v.
6 4 A	5.40		Control	0.935 v.	0.954 v.	
65	6.25		Flour Paste	0.853 v.	0.620 v.	0.233 v.
65A	6.25		Control	0.971 v.	0.965 v.	0.006 v.
66	7.20		Flour Paste	0.834 v.	0.561 v.	0.273 v.
66A	7.20		Control	0.922 v.	0.959 v.	
67	7.50		Flour Paste	0.685 v.	0.485 v.	0.200 v.
67A	7.50		Control	0.782 v.	0.836 v.	
6 8	7.90		Flour Paste	0.645 v.	0.472 v.	0.173 v.
68A	7.90		Control	0.692 v.	0.739 v.	



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Iffect of Separator Laterials on the Voltage of Manganese Dioxide When Stored in Contact at 113°F.

Separator Materials	Voltage o	Voltage of sample			
Separator materials	Initial	12 months	Reduction		
Corn Starch	0.920 v.	0.702 v.	0.218 v.		
Potato Starch	0.920 v.	0.722 v.	0.198 v.		
Hubinger's O.K. Thin Boiling #20 Starch	0.920 v.	0.692 v.	0.228 v.		
Hubinger's O.K. Waxy Sorghum Starch	0.920 v.	0.690 v.	0.230 v.		
Staley Special Battery Starch	0.920 v.	0.692 v.	0.228 v.		
P.F. Powder	0.920 v.	0.690 v.	0.230 v.		
Ground Starch Sponge	0.920 v.	0.722 v.	0.198 v.		
SD-12	0.920 v.	0.700 v.	0.220 v.		
Northern Refined Flour	0.920 v.	0.708 v.	0.212 v.		
Hallmark Powdered Potato Starch	0.920 v.	0.726 v.	0.194 v.		
Hallmark SDSL	0.920 v.	0.712 v.	0.208 v.		
Hallmark SDS	0.920 v.	0.703 v.	0.217 v.		
Hallmark Powdered Wheat Starch	0.920 v.	0.710 v.	0.210 v.		
Paygel P Wheat Starch	0.920 v.	0.708 v.	0.224 v.		
STLM	0.920 v.	0.725 v.	0.195 v.		
Shopal #8	0.920 v.	0.687 v.	0.233 v.		
Hallmark Sago Base Starch	0.920 v.	0.715 v.	0.205 v.		
Hygrade Maris	0.920 v.	0.713 v.	0.207 v.		
Saliocca without wheat	0.920 v.	0.708 v.	0.212 v.		
Saliocca	0.920 v.	0.672 v.	0.248 v		
Special Powder	0.920 v.	0.700 v.	0.220 v.		
Hubinger's O.K. Powdered Starch	0.920 v.	0.703 v.	0.217 v.		
B2455 P Wheat Starch	0.920 v.	0.711 v.	0.209 v.		
Dry Flo	0.920 v.	0.732 v.	0.188 v.		
Flogel #20	0.920 v.	0.728 v.	0.192 v.		
Nalex #15	0.920 v.	0.715 v.	0.205 v.		
Flogel #75	0.920 v.	0.722 v.	0.198 v.		
Amioca #85	0.920 v.	0.725 v.	0.195 v.		
Denatured Gluten	0.920 v.	0.759 v.	0.161 v.		
Clear Flo L	0.920 v.	0.748 v.	0.172 v.		
Santocel C	0.920 v.	0.823 v.	0.097 v.		

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elaund 9 (cont.)

Separator Laterial	Voltage of	Voltage of sample			
Sebarator wateriat	Initial	12 months	Reduction		
Special Theat Protein REE-570	0.920 v.	0.777 v.	0.143 v.		
Clear Plo H	0.920 v.	0.773 v.	0.147 v.		
Puracol R120 - 425	0.980 v.	0.735 v.	0.185 v.		
crotein #3323	0.920 v.	0.760 v.	0.160 v.		
Devitalized Wheat Gluton	0.920 v.	0.770 v.	0.150 v.		
Triticote #87	0.920 v.	0.744 v.	0.176 v.		
Special Wheat Protein R2E-1301	0.920 v.	0.750 v.	0.170 v.		
Flour	0.920 v.	0.664 v.	0.256 v.		
Soya Flour	0.920 v.	0.600 v.	0.320 v.		
Melamine Starch	0.920 v.	0.653 v.	0.267 v.		
Formalin-Urea Starch	0.920 v.	0.650 v.	0.270 v.		
Formalin' Starch	0.920 v.	0.653 v.	0.267 v.		

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FIGURE 9 (cont.)

Sample	Separator Material	Voltage	e of sam	ple afte	r stora	ge
No.	Tested	Init.	l mo.	3 mos.	6 mos.	9 mos.
23	9-D-138 Modified Tapiocca	0.920	0.768	0.760	0.751	0.731
24	Hallmark - Sago Base St. (Stein-Hall Co.)	0.920	0.792	0.772	0.755	0.731
28	Hubinger's O.K. Powdered Starch	0.920	0.790	0.772	0.754	0.723
29	B-2455 Wheat Starch (General Mills inc.)	0.920	0.791	0.772	0.759	0.725
30	Starch xR-32 (Corn Products Co.)	0.920	0.812	0.795	0.774	
31	SH-11 (Stein-Hall Co.)	0.920	0.836	0.807	0.778	
32	Clear Flo L (National Starch Co.)	0.920	0.844	0.827	0.815	
33	Clear Flo H (National Starch Co.)	0.920	0.860	0.847	0.830	
34	Dry Flo (National Starch Co.)	0.920	0.822	0.796	0.758	
35	ARD - 162 (American Maize Products)	0.920	0.833	0.814	0.786 .	
36	Pearl Corn Starch (American Maize Products)	0.920	0.832	0.809	0.786	
37	Gelex (American Maize Products)	0.920	0.828	0.816	0.802	
38	W-ll Starch (American Maize Products)	0.920	0.833	0.808	0.780	
39	Flogel 75 (National Starch Co.)	0.920	0.820	0.803	0.779	
40	Flogel 20 National Starch Co.)	0.920	0.825	0.800	0.780	
41	Nalex 15 (National Starch Co.)	0.920	0.816	0.795	0.768	

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FIGURE 9 (cont.)

Sample	Separator Material	Voltag	e of sa	mple aft	er stora	де
No.	Tested	Init.	1 mo.	3 mos.	6 mos. 9	mos.
42	Globe Fearl Starch (Corn Froducts Co.)	0.920	0.824	0.808	0.784	
43	Amigold Yellow Corn Flour (Corn Products Co.)	0.920	0.823	0.807	0.788	
44	Carboxy Methyl Starch	0.920	0.789	0.770	0.765	
45	APCG (Stein-Hall Co.)	0.920	0.810	0.804	0.798	
46	Karaya Gum (Stein-Hall Co.)	0.920	0.866	0.867	0.857	
47	Tragacanth Gum (Stein-Hall Co.)	0.920	0.810	0.792	0.773	
48	RST Starch (A.E.Staley Co.)	0.920 '	0.843	0.813	0.787	
49	1.0 Pa3 Starch (A.E.Staley Co.)	0.920	0.843	0.813	0.785	
50	O.5 Pa4 Starch (A.E.Staley Co.)	0.920	0.846	0.813	0.783	
51	Argo Brand Zein (Corn Products Co.)	0.920	0.855	0.845	0.830	
52	A-21-S Wheat Starch (General Mills Inc.)	0.920	0.805	0.795		
53	A-22 Purified Wheat Starch (General Mills Inc.)	0.920	0.806	0.799		
54	Corn Steep Liquor (A.E. Staley Co.)	0.920	0.796	0.790	0.786	
55	Special Nutrient #22 (A.E.Staley Co.)	0.920	0.798	0.793	0.787	
56	Special Nutrient #165 (A.L.Staley Co.)	0.920	0.800	0.796	0.795	

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FIGUAL	1	U

Study of Separator Materials Which Have Shown Fromise During Storage at 1600 F.

Mix For	Mix Formulation Ore/Bla		ack - 7	/1				
			24.0% NH ₄ Cl; 22.5% ZnCl ₂ ; 53.5% H ₂ O (Composition by weight)					
Storage Conditi						eliminating s stored at		
Separat	or Thickness	0.438	inch					
Ore Tes	sted	Africa	n ore					
Sample No.	Separator Mater	lal	Volta Init.	ge	of sample 2 weeks	after stor	age 6 weeks	
0	Control - Electro	olyte	0.901	V •				
1	Corn Starch		0.901	V • \	0.685 v.	No gel structure at 4 weeks		
2	Staley's Special Starch (A.E.Stale		0.901	۷.	0.725 v.	No gel structure at 4 weeks		
3	P. F. Powder (American Maize I	Prod.)	0.901	٧.	0.718 v.	No gel structure at 4 weeks		
4	SD-12 (Stein-Hall Co.)		0.901	٧.	0.714 v.	No gel st at 4 week		
5	Staley's Battery (A.E.Staley Co.)	Starch	0.901	٧.	0.717 v.	0.669 v.	0.613 v.	
6	Melamine Starch		0.901	٧.	0.672 v.	0.659 v.	0.642 v.	
7	Paygel P - Wheat (General Mills In		0.901	V •	0.713 v.	No gel stat 4 week		
8	Shopal #8 (Stein-Hall Co.)		0.901	v.	0.781 v.	No gel Structure at 4 weeks		
9	Hallmark Sago Bas Starch (Stein-Hal		0.901	٧.	0.735 v.	No gel structure at 4 weeks		
10	Saliocca without	wheat	0.901	7.	0.718 v.	No gel st at 4 week		

FIGURE 10 (cont.)

Sample	Separator haterial	Voltage	of sample	after stor	age
No.	Tested	Initial	2 weeks	4 weeks	6 weeks
11	Saliocca with wheat	0.901 v.	0.717 v.	No gel state 4 week	
12	Hubinger's O.K. Powdered Starch	0.901 v.	0.726 v.	No gel stat 4 week	
13	Globe rearl Starch (Corn Products Co.)	0.901 v.	0.717 v.	0.665 v.	No gel left
14	Flogel #20 (National Starch Co.)	0.901 v.	0.714 v.	No gel state 4 week	
15	Flogel #75 (National Starch Co.)	0.901 v.	0.713 v.	No gel state 4 week	
16	ARD-162 (American Maize Prod.)	0.901 v.	0.720 v.	No gel structure at 4 weeks	
17	Pearl Corn Starch (American Maize Prod.)	0.901 v.	0.716 v.	No gel structure at 4 weeks	
18	Polyvinyl Alcohol - high viscosity	0.920 v.	0.816 v.	0.805 v.	0.806 v.
19	Carboxy Methyl Cellulose low viscosity	0.920 v.	0.816 v.	0.810 v.	No gel left
20	Karaya Gu (Stein-Hail Co.)	0.920 v.	0.839 v.	0.823 v.	No gel left
21	Tragacanth Gum (Stein-Hall Co.)	0.920 v.	0.716 v.	0.700 v.	No gel left
22	RST Starch (A.E. Staley Co.)	0.920 v.	0.755 v.	0.661 v.	0.658 v.
23	1.0 Pa3 Starch (A.E.Staley Co.)	0.920 v.	0.753 v.	0.660 v.	0.652 v.
24	0.5 Ra4 Starch (A.E. Staley Co.)	0.920 v.	0.753 v.	0.659 v.	0.650 v.
25	Guar Gum (General Mills Inc.)	0.920 v.	0.744 v.	No gel structure at 4 weeks	
26	Argo Brand Lein (Corn Froducts Co.)	0.920 v.	0.836 v.	0.806 v.	0.764 v.

FIGURE 10(cont.)

Sample	Separator Material	Voltage of sample after storage			age
No.	Tested	Initial	2 weeks	4 weeks	6 weeks
27	Corn Steep Liquor (A.E.Staley Co.)	0.920 v.	0.795 v.	0.765 v.	0.716 v.
28	Special Nutrient #22 (A.E.Staley Co.)	0.920 v.	0.804 v.	0.775 v.	0.764 v.
29	Special Nutrient //165 (A.E.Staley Co.)	0.920 v.	0.800 v.	0.771 v.	0.764 v.
30	A-21-S Wheat Starch (General Mills Inc.)	0.901 v.	0.720 v.	0.645 v.	0.656 v.
31	A22 Furified Wheat Starch (General Mills)	0.901 v.	0.717 v.	0.653 v.	0.636 v.
32	B2455 Wheat Starch (General Mills Inc.)	0.901 v.	0.710 v.	0.650 v.	0.640 v.

FIGURE 10 (con't)

	Separator		Voltage	of sample	after store	age
Sample No.	Material Tested	Ore Tested	Initial	2 wks. at 160°F	4 wks. at 160°F	6 wks. at 160°F
32A	Nalex #15 (National Starch Company)	African ore	0.901 v.	No gel structure at 2 weeks		
33A	Flogel #75 (National Starch Company)	African ore	0.901 v.	0.713 v.	No gel stat 4 week	
34A	Waxy Maize	African ore	0.901 v.	No gel s 2 weeks	tructure a	t
35A	W-11 (Amer. Waize Products)	African ore	0.901 v.	No gel structure at 2 weeks		
36A	Gelex (Amer. Maize Products)	African ore	0.901 v'.	No gel structure at 2 weeks		
37A	ARD-162 (Amer. Maize Products)	African ore	0.901 v.	0.720 v.	No gel s at 4 week	
38A	Pearl Corn Starch	African ore	0.901 v.	0.716 v.	No gel sat 4 week	
39A	Control (Electrolyte only)	Baker's Anal. MnO ₂	0.874 v.	0.855 v.	0.849 v.	0.831 v.
40A	Corn Starch	Baker's Anal. MnO ₂	0.874 v.	0.645 v.	0.611 v.	0.600 v.
41A	Potato Starch	Baker's. Anal. MnO2	0.874 v.	0.647 v.	0.632 v.	0.520 v.
42A	Flour	Baker's Anal. Mn02	0.874 v.	No gel s 2 weeks	tructure a	t

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FIGURE 10(con't)

	Separator		Voltage o	of sample	after store	fter storage		
Sample No.	Material Tested	Ore Tested	Initial	2 wks. at 160°F	4 wks. at 160°F	6 wks. at 160°F		
43A	Control (Electrolyte only)	Burgess Chem.	1.010 v.	0.943 v.	0.927 v.	0.898 v.		
44A	Corn Starch	Burgess Chem.	1.010 v.	0.632 v.	No gel sat 4 week			
45A	Potato Starch	Burgess Chem.	1.010 v.	No gel structure at 2 weeks				
46A	Flour	Burgess Chem.	1.010 v.	No gel structure at 2 weeks				
47A	Control (Electrolyte only)	Burgess Electro	1.055 v.	1.001 v.	0.961 v.	0.943v.		
48A	Corn Starch	Burgess Electro	1.055 v.	0.646 v.	No gel structure at 4 weeks			
49A	Potato Starch	Burgess Electro	1.055 v.	No gel s 2 weeks	tructure a	t		
50A	Flour	Burgess Electro	1.055 v.	No gel s 2 weeks	tructure a	t		
51A	Control (Electrolyte only)	Crypt. (Lt. Hyd.)	0.944 v.	0.837 v.	0.814 v.	0.801 v.		
52A	Corn Starch	in ZnCl ₂ solution	0.944 v.	0.640 v.	0.612 v.	0.535 v.		
53A	Potato Starch		0.944 v.	0.632 v.	No gel s			
54A	Flour		0.944 v.	0.632 v.	No gel s at 4 weel			

PART IV

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PAPER COATED

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Part IV - Paper Coated Separators

Many studies were conducted in this project to determine what effect a conventional separator material has on the voltage reduction of manganese dioxide. Considerable data have been collected to prove that a conventional separator substantially reduces the voltage of a manganese dioxide ore. Of the various types of materials tested, proteins, glutens and most gum type materials caused the smaller voltage reduction.

However, as this class of material cannot be handled in the conventional manner (due to the lack of gel formation), considerable effort was put on the study and use of paper-coated separators. Is this study has many different and unique features it is reported here separately in order to clearly establish this particular work.

The preliminary step in this new phase of work in separators was to order a paper coating machine. A paper gumming machine which can be adapted for coating paper was obtained from the New Jersey Machine Corporation of Hoboken, New Jersey. The machine did not arrive until May 7, 1953 which caused considerable delay in the accomplishment of this work. In the meantime, backer ground work was started in regard to this investigation by fabricating and testing cells made from Eveready methyl cellulose paper as a separator.

With the arrival of the machine, preliminary trial runs were made in order to establish sound procedures in regard to coating the paper with the separator material. A roll of Nibroc Kraft Water Paper was obtained in which to apply the various materials. It was found that two coatings applied a film slightly over 1001* giving a combined paper thickness of .001*.

As soon as familiarity with the coating machine was achieved, paper was coated using Carboxy Methyl Cellulose Gums, Polyvinyl alcohol and various other gum products. The materials were prepared for coating by dissolving in distilled water in percentages ranging from 1% to 10% by weight until a fairly fluid solution was prepared. The solutions were desired in this fluid state to allow even flow and thickness when applied to the paper. The paper when dried was then cut into squares 31 x 4 x, wrapped on a hand mandrel, the bottom Tolded over, and lined into the zinc cans for fabrication into dry cells. The paper was inserted into the cans by the use of liner mandrels approximately 1-7/64 diameter.

Certain problems were encountered initially in regard to the Mabrication of paper lined cells. Basically they are as follows:

- (a) Type of can construction and seal to be used.
- (b) Method of tamping mix and the addition of the depolarizer to the paper-lined cans.
 - (c) Proper amount of mix weight desirable in this type of cell.

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(d) Proper degree of mix wetness satisfactory for storage at elevated temperatures.

In regard to the problem of can construction, it was finally decided that a standard leakproof assembly would provide the most optimum conditions for this type of work. This consists of a flat-bottom zinc leakproof can with a high resistant polystyrene cap and a polyethylene gasket as a seal. The gasket was stamped out of polyethylene sheets approximately $1\frac{1}{2}$ in diameter. Several new tools were required in order to insure proper specifications for the leakproof cell. These tools consisting of a tamping rod, pencil guides and liner rods were made of a hard rubber composition.

After several experimental runs it was decided that the best procedure was to tamp a mix slug of from sixty to sixty—five grams, 2½ long by 1.029 in diameter. These slugs were then placed in the previously lined cans and retamped with a tamping rod which is 1.113 in diameter. An adjusted stop on the arbor press regulated the final core height at 1-5/8.

The carbon rod was inserted into the depolarizer with the use of a pencil guide which measures 1.131 in diameter. Finally, the excess paper was folded down and pressed over the depolarizer and around the carbon rods by exerting slight pressure on the pencil guide. The small compression brings contact between the depolarizer, separator and zinc can by squeezing electrolyte out of the mix, and wetting the paper separator which results in proper conductance within the cell. Complete specifications for the various tools and materials used are reported in Figure A along with a chart showing the percentage of mix ingredients for an average mix.

The electrolyte that has been used in all testing to date is standard battery electrolyte (zinc chloride, ammonium chloride and water) with 0.25% mercuric chloride added.

The most difficult problem to overcome in the construction of paper lined cells concerns the proper degree of mix wetness. Considerable emphasis must be placed on this condition because of its extreme importance toward dry cell efficiency especially at elevated temperatures. To understand what occurs over a varied series of mix wetnesses, cells were constructed using mixes that ranged from extreme dryness to extra wetness. The table in Figure #1 shows capacities received from these cells. The first batch of cells in the series gave no results at two weeks at 160°F, because of their extreme dryness. As the mix wetness increases, it can be noted that capacities increase, but this is accordingly accompanied by an increase in corrosion of the cells. Subsequent results have shown that the medium wet mix (approximate average regular dry mix) is the best mix condition for paper lined cells.

Capacities obtained to date on cells fabricated are reported in Figure #2. Several exceptional facts are evident from this data. Eveready methyl cellulose paper has been outstanding as a paper separator giving capacities that far exceed the conditions specified in the contract. The other materials

SECURITY INFORMATION RESTRICTED tested have not performed satisfactorily. At elevated temperatures capacities are completely gone. This most probably is attributable to the fact that these materials completely break down at these high temperatures thus destroying the efficiency of the cell.

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Conclusions

- 1. Most water soluble materials that show promise as separator materials can be readily coated for paper-lined cells.
- 2. Close regulation of the mix wetness must be maintained in order to achieve the optimum conditions of capacity and yet overcome the problem of corrosion.
- 3. Except for Eveready paper, the materials tested to date have completely broken down at elevated temperatures thus becoming useless in the cell as a separator.
- 4. Eveready methyl cellulose paper has shown exceptional merit as a separator material and cells made with this paper have given capacities far exceeding the specifications in the contract for temperatures of 130°F. = 50% R.H. and 160°F. 50% R.H.

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TABLES AND RESULTS

PART IV

PAPER COATED SEPARATORS

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FIGURE A

Specifications of Various Tools and Materials Used in Producing Paper Lined "D" Cells (Paper Coated Separators)

					
Tools @ Materials	Composition	Diameter	Length	Weight	Thickness
Coated paper	Paper-Nibro Craft Water	****	3 ¹ 4"x 4"	0.60 gm.	•00H#
Liner mandrels	Hard rubber	1.062"	63/4"	# w # w &	# m m m m
Flat bottom leakproof Can	Zine	1.250"	2.250"	14 gms.	wall .016"
Core mold	Stilite plated steel	1.058"	4 4 "		wall •125"
Tamping rod (mold)	Stilite plated steel	1.045"	5불"		
Mix slug	Depolarizer material	1.029 (approx)	24"	60-65 gms	
Final core slug diminsion	Depolarizer material	1.210" (approx)	15/8"	60-65 gms	
Tamping rod (can)	Hard rubber	1.113"	74季4		
Pencil guide	Lucite	1.131"	1ੇਹਾਂ		hole dia
Polyethylene gasket	Polyethylene	1 à "		0.20 gms	•005"

Percentage of Mix Ingredients (average mix)

African Ore	64.4 %
Carbon Black	9•2 %
Sal (NH _L Cl)	9•2 %
Standard Electrol- yte (0.25% HgCl2)	17.2 %

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FIGURE 1

Construction of Paper Lined Cells ("D" Size) in the Varied Wetness Series

Lot No.	Mix Formulation	Mix We tness	Separator	Subseal	Seal
D-1280	100% African Ore 7/1 ore to black Inhibitor present (•25% HgCl ₂)	Extreme dry	Eveready Methyl Cellulose Paper	Poly- ethylene gasket	Poly- Styrene cap
D -1 283.	100% African Ore 7/1 ore to black Inhibitor present (•25% HgCl ₂)	Regular "D" cell wetness	Eveready Methyl Cellulose Paper	Poly- ethylene gasket	Poly- Styrene cap
D-1282	100% African Ore 7/1 ore to black Inhibitor present (.25% HgCl ₂)	Medium wetness	Eveready Methyl Cellulose Paper	Poly- ethylene gasket	Poly- Styrene cap
D-1281	100% African Ore 7/1 ore to black Inhibitor present (.25% HgCl ₂)	Extreme wetness	Eveready Methyl Cellulose Paper	Poly - ethylene gasket	Poly- Styrene cap

FIGURE 1A

Capacity of Paper Lined Cells ("D" Size) in the Varied We tness Series

	Capacity Results								
	BA 30 (days)	BA 8 (1	nou rs)	BA 408/U (hours)				
Lot No•	Initial	2 weeks at 160°F	Initial	2 weeks at 160°F	Initial	2 weeks at 160°F			
D-1 280	14.9 days	Cells no good	208 hours	Cells no good	25.2 hours	Cells no good			
D-1283	18.1 days	U ₁ .1 days	269•2 hours	203.3 hours	28.3 hours	16.8 hours			
D-1282	19.6 days	15•3 days	284 hours	202.6 hours	28.7 hours	18•l4 hours			
D-1281	21.5 days	15.6 days	292.8 hours	2[12.3 hours	32.8 hours	22.8 hours			

FIGURE 2

Paper Lined "D" Cells For Storage (Construction).

	ge Time Before Initial ng At 70° F.	2 weeks at 70		
Stora	ge Time For Cells Store (Tested at 70° F.)	3 months at 7 3 months at 1 3 months at 1 2 weeks at 1	13°F - 95%	R.H.
Lot No.	Mix Formulation	Separator	Subseal	Seal
D1003	100% African Ore 7/1 - ore/black 0.25% HgCl2 -(inhib	Eveready Methyl Cellulose Paper	Poly- ethylene gasket	Poly- styrene cap
D1284	100% African Ore 7/1 - ore/black 0.25% HgCl2-(inhib)	Eveready Methyl Cellulose Paper	Poly- ethylene gasket	Poly- styrene cap
D1112	100% Western Elect. 7/1 - ore/black 0.25% HgCl2-(inhib)	Eveready Methyl Cellulose Paper	Poly- ethylene gasket	Poly- styrene cap
D1172	30% African Ore 70% Western Electro 7/1 ore/black 0.25% HgCl2-(inhib)	Eveready Methyl Cellulose Paper	Poly- ethylene gasket	Poly- styrene cap
ב84עם	100% African Ore 7/1 ore/black 0.25% HgCl2-(inhib)	Carboxy methyl Cellulose Paper (low viscosity)	Poly - ethylene gasket	Poly- styrene cap
D2294	100% African Ore 7/1 ore/black 0.25% HgCl2-(inhib)	Carboxy methyl Cellulose Paper (medium viscosity)	Poly- ethylene gasket	Poly- styrene cap
D1793	100% African Ore 7/1 ore/black 0.25% HgCl2=(inhib)	Carboxy methyl Cellulose Paper (high viscosity)	Poly- ethylene gasket	Poly- styrene cap
D2309	100% African Ore 7/1 ore/black 0.25% HgCl2-(inhib)	Polyvinyl alcohol "Elvanol"	Poly- ethylene gasket	Poly- styrene cap

FIGURE 2 (Cont.)

Lot No.	Mix Formulation	Separator	Subseal	Seal
D2337	100% African Ore 7/1 ore/black 0.25% HgCl2-(inhib.)	Polyvinyl alcohol "Elvanol" (medium viscosity)	Poly - e thylene gaske t	Poly- s tyre ne cap
D2390	100% African Ore 7/1 ore/black 0.25% HgCl2-(inhib.)	Arabic Gum "Acacia"	Poly- ethylene gasket	Poly- styrene cap
DS 101	100% African Ore 7/1 ore/black 0.25% HgCl2-(inhib.)	Locust Bean Gum "Italian"	Poly- ethylene gasket	Poly- styrene cap
D2580	100% African Ore 7/1 ore/black 0.25% HgCl2-(inhib.)	Swifts Colloid Gum (No. 1)	Poly- ethylene gasket	Poly- styrene cap
D25 79	100% African Ore 7/1 ore/black 0.25% HgCl2-(inhib.)	Gelatine Gum	Poly- ethylene gasket	Poly- styrene cap

FIGURE 2A

Paper Lined "D" Cells For Storage (Capacity Results)

TEST RESULTS

TEST RESULTS							
Book	Snaacaa	Storage Temperature					
No.	Specified	70 [°]	F.	113 ⁰ F.	130° F.	160° F.	
140 •	Test.	Initial	3 months	3 months	3months	2 weeks	
	BA 30	20.7	18.3	18.9	17.9	15.9	
	(days)	() • ()		100)	±(•)	±)•)	
p1003	BA 8	283	274	251	241	263	
	(hours)		- (+		<u></u>		
	BA 408	27.6	32.4	26•2	26.6	25 •4	
	(hours))—F		2000	-) •+	
	BA 30	21.0	18.5	17•9	17.0	17.7	
	(days)				<u> </u>	<u> </u>	
D1 284	BA 8	298	288	2 88	249	280	
}	(hours)						
	BA 408	3 2∙႘	33•2	28.9	30.4	28.3	
	(hours)						
	BA 30	34.5	21.6	21.3	-22.3	1 9 . 5	
•	(days)						
D1112	BA 8 (hours)	510	430	38 3	486	458	
	ва 408						
	(hours)	61.7	67.7	65•5	63.5	村1.0	
	BA 30						
	(days)	33•3	28.6	32.1	31.6	19.2	
	BA 8	1 25					
D1172	(hours)	45 8	400	447	419	336	
	BA 408		5 0 1	/0.5		100	
ļ	(hours)	5 7• 9	59•4	60.7	57.1	40.0	
	BA 30	3 c ^	7)	10 0	10.0	Cells no	
	(days)	15.0	บ₁•5	12.2	10.2	good	
D11481	BA 8	254	245	205	1 20:	Cells no	
Differ	(hours)	<i></i>	247	205	124	good	
	ва 408	26.9	19.•9	υ _{1.} .7	14.3	Cells no	
·	(hours)		-77	****** [good	

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FIGURE 2A (Cont.)

TEST RESULTS

 	·	TEST RESULTS							
Book No•	Specified Test	Storage Temperature							
		70° F.		113° F.	130° F.	160° F.			
		Initial	3 months	3 months	3 months	2 weeks			
D2294	BA 30	71. 0				Cells no			
	(days)	14.0	,		315 (S. d.	good			
	BA 8	269				F0 F			
	(hours)					52.5			
	BA 408	23.7				Cells no			
	(hours)					good			
D17 <u>9</u> 3	BA 30	15.1	11.1	3.0	2.2	Cells no			
	(days)		11.1)••		good			
	BA 8	240	169	125	90.1	16.0			
	(hours)								
	BA 408	26.9	22•3	17.3	Cells no	Cells no			
	(hours)				good	good			
D2309	BA 30	16.6				Cells no			
	(days)	2000				good			
	BA 8	283	,			53			
	(hruis)								
	BA 408	2 8.6	,			14.7			
	(hours)					·			
D2337	BA 30	15.5				Cells no			
	(days)					Good			
	BA 8	275				99			
	(hours)								
	BA 408	26.6				Cells no			
	(hours)					good			
D2390	BA 30	12.7 241				Cells no			
	(days)					good			
	BA 8					56			
	(hours)								
	BA 408	26.2		•		Cells no			
	(hours)					good			

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FIGURE 2A (Cont.)

TEST RESULTS

ــــــــــــــــــــــــــــــــــــــ		TEST RESULTS						
Book No.	Specified Test	Storage Temperature						
		70° F.		113° F.	130° F.	160° F.		
		Initial	3 months	3 months	3 months	2 weeks		
D2401	BA 30	9•3				Cells no		
	(days)					good_		
	BA 8	255				Cells no		
	(hours)					good		
	BA 408	26.9				Cells no		
	(hours)					good		
D2580	BA 30	6.8				Cells no		
	(days)					good		
	BA 8	227				Cells no		
	(Hours)					good		
	ва 408	16.4				Cells no		
	(hours)					good		
D2579	BA 30	10.7			İ	Cells no		
	(days)					good		
	BA 8	ं 2214				Cells no		
	(hours)					good		
	BA 408	25.1				Cells no		
	(hours)					good		

-*** Blank spaces in this data are due to incomplete returns of capacity results.

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PART V

POLARIZATION STUDY

CORROSION INHIBITORS

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Part V - Polarization Studies

A. Polarization Inhibitors

A corrosion inhibitor in order to be acceptable for use in a dry cell must prevent corrosion of the zinc anode and function without interfering with the electrochemical reactions of the cell. A material which produces a high anodic polarization must not cause anodic passivity of the zinc. The inhibitor should be capable of inducing an appreciable degree of cathodic polarization over the whole metal surface to provide an effective corrosion barrier. The corrosion barrier is defined as the algebraic difference between the cathodic and anodic voltages.

Extensive experimental work was done to evaluate inhibitors suitable for the high temperature conditions imposed by this contract. Anodic and cathodic polarization studies were made after various periods of storage at 113°F, 130°F and 160°F.

The polarization apparatus and methods employed for the study are described in Part I (Equipment - Procedure) and a diagram of the operations is shown in Figure #1 of that section.

Use of this apparatus permitted the investigation of anodic inhibitors apart from the complicated reactions occurring in the cathode area.

These polarization studies were divided into two groups: (1) studies in which a current density equivalent to that of the BA-30 drain (4.33 ma/cm²) is imposed on the electrode immediately and held constant while the voltage changes are recorded with time, and (2) studies in which extremely light current densities are imposed initially and increased by small increments during the run. In the second instance, time is allowed for voltage (or polarization) equilibrium to develop before the voltage readings are recorded. Voltage is then plotted against current density. The change of cathodic polarization with current density is measured alternately with that of the anodic polarization by using two identical zinc electrodes in conjunction with a calomel reference electrode.

Some forty-two inhibitors have been analyzed on both these tests. The results of six representative samples for anodic polarization of zinc are tabulated and shown graphically in Figure #1. The majority of the samples tested exhibited a large amount of corrosion over the six month period which is partly due to the excess of electrolyte present as compared to the amount of zinc. Those samples which showed a small amount of corrosion after six months indicate very good inhibitive powers (small concentrations of sodium dichromate and Antaron R-155). Sodium dichromate produces a heavy film on the surface of the zinc but when this film becomes too excessive passivation of the zinc will occur as is shown in the Figure 1B. Figure 1C shows the effects of devitalized wheat gluten which is representative of all the glutens tested, while Figure 1D displays the action of special wheat protein R2E-570 which represents the proteins tested. Antarox A-403 was chosen to represent the various wetting agents and detergents used and is shown in Figure 1E.

. . . .

Of the various organic compounds analyzed as inhibitors, Quinaldine appears to give the best inhibitive action and is reported in Figure 1F.

It can be noticed in all of the polarization samples that after storage for extended periods of time, the zinc tends to show signs of passivity. In the case of sodium dichromate the passive tendency is not overcome, whereas in the case of special wheat protein R2E-570 the effect is not so strong as to render the zinc passive. These experiments do not give a clear indication of the barrier which any inhibitor will set up to prevent corrosion. They do indicate quite clearly whether any inhibitor causes passivity. If a material causes passivity it will give poor battery performance since it prevents the discharge of zinc ions. Anodic polarization data were obtained on a given sample initially and after one month storage at 113°F. A second sample stored for one month at 113°F was also tested in the same manner. This was done in order to determine if initial polarization readings affected the delayed readings. In all cases, both samples read after one month storage at 113°F gave values which closely approximate each other.

In all these samples, a slight amount of potato starch was added to the electrolyte (l gram/40 mls) to partially immobilize it and prevent creepage of the solution up the electrodes, especially samples where wetting agents were used an inhibitors.

It is interesting to note that the samples which were tested using both unamalgamated and amalgamated zinc shows that the latter maintained a higher voltage throughout the polarization period. This should be expected because the voltage of the amalgamated zinc is normally higher than that of unamalgamated zinc. Yet anodic-cathodic polarization results of amalgamated and unamalgamated zinc show the slight corrosion barrier exhibited by amalgamated zinc (Figure #2). On this basis it would be expected that amalgamated zinc corrode, badly during storage.

This was found to be especially true of amalgamated zinc when stored in electrolyte for three months at 113°F. Amalgamated zinc undergoes a weight loss five times greater than the chrome inhibited zinc and ten times greater than the zinc inhibited with Protein R2E-570, Antaron R155 or like materials.

This is not to say that amalgamation of zinc does not in some respect inhibit corrosion. The mercury does tend to level the potential of anodic and cathodic areas in the zinc. It also raises the hydrogen overvoltage making it more resistant in acid solutions. However, there is no barrier set up to prevent corrosion and if there is a tendency for any area to become either cathodic or anodic, corrosion will proceed with very little resistance in terms of polarization barriers. Results obtained from the experiments indicate that with amalgamated zinc in particular, discharge other than hydrogen ion discharge takes place without difficulty. It is quite possible therefore that hydrogen evolution is of secondary nature in the corrosion of amalgamated zinc. The first step would be the direct discharge of zinc which would in turn react with the electrolyte to liberate hydrogen.

The second part of the experiments to evaluate inhibitors involves the measurement of both anodic and cathodic polarization at low current densities. The result of this work determines the ability of inhibitors to prevent corrosion and the amount of corrosion barrier exhibited. The experimental procedure is similar to anodic polarization and the various alterations are explained in Part I (Equipment-Procedure).

With this arrangement it is possible to measure the cathodic and anodic voltage of the zinc in the same corrosion solution under any desired polarization current. In these first experiments the polarization current was vaired from a low value (0.08 ma) to a value approximating the BA-30 drain (3.5 ma). Very low polarization currents were used since the corrosion current of a battery while on shelf is very low. The higher this current becomes the faster will be the rate of corrosion. The results of nine materials tested in this manner at temperatures of 70°F, 113°F and 160°F are tabulated and shown graphically in Figure #3. These data show that some inhibitors cause very little change in either cathodic or anodic voltage as the current density is increased. Others show very rapid change in the anodic or cathodic voltage or both with a small increase in polarization current during the initial portion of the test.

For corrosion of batteries on shelf the initial parts of the curves are all that will be discussed as the polarization current is increased beyond one milliampere, conditions approximate those of battery discharge and the curves indicate to what extent an inhibitor passivates the zinc.

An ideal inhibitor would give a sharp and rapid rise in the cathodic voltage and a rapid but moderate decrease in the anodic voltage. The inhibitor most closely approximating this in the series is Antaron R-155 (Figure 3C). This inhibitor gives a sharp increase in the cathodic voltage and a sharp decrease in anodic voltage with only a slight increase in the polarization current. Yet the overall decrease in the anodic voltage is not sufficient to indicate serious passivity.

The rapid divergence of the anodic and cathodic voltage curves is desired since it is the difference between the values of the anode and cathode at polarization current drains which give rise to a corrosion barrier.

The sample containing sodium dichromate (Figure 3a) inhibitor exhibit a higher cathodic voltage than does Antaron R-155 but this high voltage is not reached so rapidly. Also the anode polarization is small at low current drains. A second undesirable feature is the fact that the anode voltage of chrome inhibited samples continues to decrease as the current is increased instead of leveling out and this indicates that it will passivate zinc to some degree.

When Antaron and chrome (Figure 3h) were used jointly, the cathodic curve is improved over that of pure chrome but there is little effect on the anode voltage.

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The use of Quinaldine (Figure 3i) as a corrosion inhibitor proved somewhat satisfactory. In the initial part of the curve, a rapid rise in the cathodic voltage occurs with only a small amount of anodic polarization. As the current increases the tendency toward passivation is not too pronounced indicating good inhibitor qualities.

Antarox A-403, Protein R2E-570, and denatured gluten give curves similar to those obtained with Antaron R-155 and Antaron with mercury but the anodic and cathodic polarization values are not as great.

On the basis of the polarization studies the following working hypothesis was evolved for samples undergoing simulatenous cathodic and anodic polarization and was used in evaluating inhibitors.

(1) An ideal inhibitor will have (a) a maximum difference between the initial open circuit voltage and cathodically polarized voltage at very low current densities, (b) a moderate anodic polarization and no evidence of gas evoluation on shelf.

On the basis of anodic polarization (voltage vs. time) the following working hypothese were developed:

- (1) Fours types of anodic polarization curves have been evidenced:
 (a) a curve showing high initial polarization with poor anodic recuperation throughout discharge, (b) a curve showing high initial polarization with rapid anodic recuperation during the discharge, (c) a curve showing very low initial polarization with little or no increase in anodic voltage during discharge, (d) a curve showing very high polarization with no recuperation (passivation).
- (2) Type 1(a) indicates either very thick films or very tenacious films. They may show low corrosion but will give poor battery performance.
- (3) Type 1(b) indicates a thin film which is easily removed by an anodic current. If initial anodic polarization is not too high it should give good battery performance.
- (4) Type 1(c) will give excellent battery performance but corrosion will be bad unless the cathode polarization value is high.
- (5) Type 1(d) indicates little corrosion will occur but destroys battery performance. If corrosion does occur it will be confined to small pits.

Figure #4 is presented to illustrate some of the various divergent curves encountered in this polarization study.

Figure #5 is a condensed graph showing the effect of chrome concentration on the corrosion barrier over various temperatures. One percent

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sodium dichromate appears to be the most optimum percentage. In polarization studies, sodium dichromate was used as the source of chromate ions but it was desired to eliminate the sodium ion since it was not known what effect it had in the battery. A polarization series was run to compare the effect of sodium and ammonium dichromate and very little difference was observed in the anodic and cathodic values of the two inhibitors at any given percentage. Therefore, it was concluded that the polarization results obtained in the past using sodium dichromate would also apply for ammonium dichromate.

As a result a comprehensive comparison between ammonium chromate and ammonium dichromate was made to find which supplies the best inhibiting qualities. Polarization results of a range from 0.1% to 3.0% were obtained. Figure #6 present several of the tables and graphs for various percentages over this range. In the figures anodic polarization (in which voltage is plotted against time) is shown in the figures with the suffix A and simultaneous cathodic and anodic polarization in which voltage is plotted against polarization current is shown in the figures with the suffix B. It should also be pointed out that the cathodic and anodic polarization studies were run at a much lower current density than was the rule in previous experiments. It is felt that the range covered in these studies (0.01 ma to 0.3 ma) is of more interest in defining the inhibiting properties of any material because it more closely approximates the average corrosion current which is present in a battery when the battery is on shelf. The corrosion current will vary from cell to cell but under normal conditions (excluding internal shorts, elevated temperatures or excessive paste line or bottom washer corrosion) will fall within the current range used for these experiments.

These polarization studies were conducted at both 70°F and 160°F. In both cases the samples were allowed to stand for twenty-four hours so that equilibrium might be reached. The following general conclusions were reached based on the studies at the two temperatures.

- (1) The O.C.V. of the sample is depressed by an increase in temperature.
- (2) The tendency toward passivity is increased by increasing the temperature as shown by the anodic polarization curves where voltage is recorded with time.
- (3) The amount of cathodic polarization is decreased by an increase in temperature.
- (4) The corrosion barrier (difference between anodic and cathodic voltage at any given current density) is decreased by an increase in temperature.

Specific conclusions which were reached regarding the relative merits of ammonium dichromate and ammonium chromate as inhibitors are as follows:

(1) Little difference was observed in the cathodic-anodic curves of

these two below 1.0%.

- (2) Above 1.0% the following was true of the anodic-cathodic polarizations (a) cathodic polarization at 70°F due to the chromate is approximately twice that due to the dichromate, (b) anodic polarization (at various currents) at both 70°F and 160°F due to the chromate is approximately half that of the dichromate, which indicates that the chromate has less of a tendency for passivity, (c) curves of anodic polarization versus time also indicate that the chromate cause less polarization at concentrations above 1.0%. Below 1.0% the curves are very similar.
- (3) On the basis of the initial data shown in this report, 2.0 and 3.0% (NH₁) CrO appear to be the most promising concentrations for inhibitors in this series, exhibiting the desired characteristics of low anode polarization and high cathode polarization. This is especially true at room temperature (70°F) .

The system of running polarization studies at a much lower current density (0.01 ma to 0.3 ma) was continued in further experimentation of inhibitors. Tests were made using various combinations of inhibitors, including mercury and chrome, chrome and Antarox A403, mercury and Antrox A403, chrome and Antaron R155, mercury and Antaron R155, chrome and Neutronyx, and mercury and Neutronyx. The individual components of these combinations were tested as a basis of comparison. The results of these studies are shown in Figure 7. It should be pointed out that Antaron R155, Antarox A403 and Neutronyx fall in the category of wetting agents and detergents. Antaron R155 and Antarox A403 are obtained from Antara Products while Neutronyx is supplied by the Onyx Oil and Chemical Company.

On the basis of anodic polarization only, where voltage is plotted versus time at a constant current density of 4.33 ma/cm², the following conclusions were drawn.

- (1) Mercury by itself (amalgamation process) gives very little anodic polarization. When a current is imposed on the zinc electrode a slight drop in voltage is evidenced immediately and the voltage then levels out and remains relatively constant. This is true of samples tested at both 70°F and 130°F.
- (2) A combination of mercury and ammonium chromate gives a curve similar to that obtained from ammonium chromate alone. However, the initial voltage drop is not as severe as would be expected were the chromate the only inhibitor. At 130°F a tendency toward passivity is exhibited by this combination (See Figure 7).
- (3) Antaron R155 and Antarox A403 show satisfactory anode polarization curves at 70°F. That is, the initial drop is not too severe and voltage recuperation takes place at a relatively rapid rate. At 130°F both materials show only a slighty voltage drop and little recuperation. However, satisfactory

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results should be obtained during discharge even at elevated temperatures (See Figure 7a).

- (4) Combinations of mercury and Antaron and of ammonium chromate and Antaron show good anode polarization curves at 70°F. At 130°F the combination of mercury and Antaron exhibits satisfactory anode polarization while the initial voltage decrease when using ammonium chromate and Antaron is excessive and low amperage will result (See Figure 7b).
- (5) Combinations of ammonium chromate and Antarox ALO3 and ammonium chromate and Neutronyx show nearly the same anodic polarization curves as the combination with Antaron RI55 with the exception that the curves obtained are better after high temperature storage.
- (6) Combinations of mercury and Antarox A403 or mercury and Neutronyx exhibit curves similar to that obtained from mercury alone indicating that the Antarox A403 and Neutronyx take little part in the polarization process.

On the basis of simultaneous anodic and cathodic polarization where voltage is plotted against current densities, the following conclusions were drawn.

- (1) The use of mercury as the only inhibitor gives a very slight corrosion barrier (difference between anodic and cathodic voltage at any given current density). However, in the case of mercury this does not mean that corrosion will take place rapidly since polarization is based mainly on film formation and the amalgamation process is unique in itself as previously explained (See Figure 7).
- (2) Combinations of mercury and Antarox ALO3 and mercury and Neutronyx also show a very small corrosion barrier indicating as stated previously that these wetting agents take little part in the polarization process.
- (3) A combination of mercury and ammonium chromate yields a curve falling approximately half way between the curves of these materials when used alone. These curves appear to have properties due to both the mercury and the chromate.
- (4) Combinations of ammonium chromate and Antarox A403, Antaron R155 or Neutronyx give very good polarization curves and a relatively high corrosion barrier. This is especially true at 70°F and also holds for 130°F. The same conclusion is also reached as the result of a study of the curves exhibited by Antarox A403 and Antaron R155 when used separately with the exception that Antarox A403 exhibits only fair curves at 130°F.
- (5) The best polarization results exhibited in this series is obtained from a combination of mercury and Antaron R155. The curves for this combination are shown in Figure 7b. The polarization for this combination at 70°F is ideal, based on the hypothesis given early in this section. Excellent polarization

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is also exhibited by this combination when tested at 130°F.

In the above series the samples were allowed to stand for twenty-four. hours, at the required temperature in order for equilibrium to be attained.

In all cases, with the exception of mercury, the percentage given was by weight of the electrolyte used. Where mercury was used the percentage of mercury involved was calculated from the area of the zinc electrodes.

B. Inhibitors and Corrosion Studies

Visual corrosion studies have been conducted to substantiate data already obtained on various inhibitors from polarization studies. In these experiments, strips of zinc were stored in electrolytes containing various inhibitors in different percentages. Two strips of zinc were included in each sample, one of which was completely immersed in the electrolyte solution while the other was only partially immersed. The latter strip was included to study the corrosion which occured at the air line. These samples were stored at 113°F and inspected and weighed periodically. Data obtained from these studies are shown in Figures 8 (zinc partially immersed) and 9 (zinc totally immersed). These data include the weight of the zinc strip initially and after twelve months storage at 113°F and also the percentage of weight loss during this period. In cases where complete corrosion occured at the air line, the data have not been included because of the inaccuracy involved.

The results obtained from these studies coincide very well with those obtained from polarization studies. The dichromate film gives by far the best inhibition of the materials tested. As shown in the polarization studies 1.0% sodium dichromate appears to be the most optimum percentage (see Figure 5). However, the polarization studies indicated a very substantial degree of passivity with this percentage and hence it is felt that best results on the basis of good inhibition and capacity can be obtained using 0.3 to 0.5% sodium dichromate by weight of electrolyte in the separator.

Good results were also obtained from Antaron R-155 and some of the proteins especially Special Wheat Protein R2E-570 and R2E-1301 which were obtained from General Mills, Inc.

With the exception of those samples containing mercury, corrosion over the zinc surface was quite even. Some pitting was noted in the samples having Sequestrene NA2 and NA4 as the inhibitor and a heavy pitting type attack was observed in the amalgamated sample.

As stated previously the chromate process for inhibiting in cells stored at high temperatures appear to be quite satisfactory whereas the amalgamation process does not.

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Conclusions

- 1. An ideal inhibitor for battery application should cause zinc to exhibit high cathodic polarization and moderate anodic polarization.
- 2. The amalgamation of zinc tends to equalize the voltage of the anodic and cathodic areas on the zinc surface. However, no appreciable corrosion barrier is exhibited by amalgamated zinc and any variation in potential on the surface will cause corrosion. This corrosion process will proceed with little impedance.
- 3. Corrosion of amalgamated zinc does not appear to involve a direct liberation of hydrogen. The corrosion process probably involves a discharge of free zinc, which because it is a finely divided and active form, reacts directly with the electrolyte to liberate hydrogen. This hypothesis is based on the fact that the low cathode polarization does not seem to be compatible with the high hydrogen overvoltage of amalgamated zinc as recognized in literature.
- 4. Weight loss measurements show dichromate, Antaron R-155 and protein materials to be the best inhibitor materials tested to date.
- 5. It is not possible to obtain as good inhibition at elevated temperatures as can be obtained with the same material at room temperature.
- 6. Polarization studies of ammonium chromate and ammonium dichromate show the former to be superior as an inhibitor especially at the higher percentages. Percentages of 2.0% and 3.0% ammonium chromate by weight of electrolyte gave excellent initial results.
- 7. A combination of mercury and Antaron R-155 as an inhibitor gave an ideal anodic-cathodic polarization curve. This combination should give very good inhibitor properties especially at room temperature.

TABLES AND GRAPHS

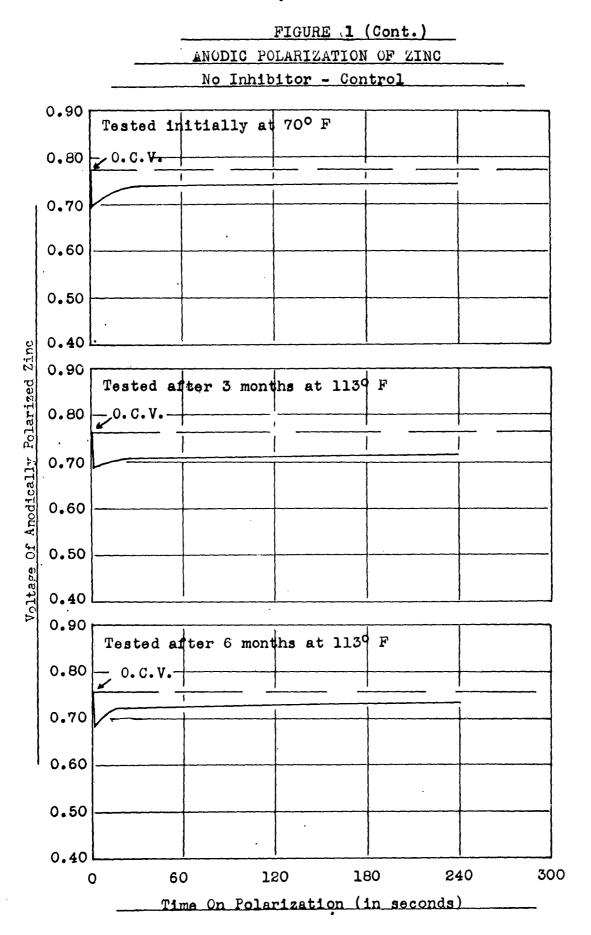
PART V

POLARIZATION STUDIES - CORROSION INHIBITORS

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FΤ	GI	ΤR	FC.	.7	

Series No.	1				
Type of sample	Electrolyte - Pote Inhibitor - None,	Electrolyte - Potato Starch (40 mls/l gr) Inhibitor - None, Unamalgamated zinc only			
Anodic Polariza	tion Current Densi	ty 4.33	3 ma/sq.cm.		
	Reading taken initially at 70° F.	Reading taken after 3 months at 113° F.	Reading taken after 6 months at 1130 F.		
O.C.V. before polarization	0.775 v.	0.761 v.	0.755 v.		
Time on polarization		Anodic Voltage			
l second	0.698 v.	0.688 v.	0.68 3 v.		
15 seconds	0.728 v.	0.702 v.	0.721 v.		
30 seconds	0.738 v.	0.706 v.	0.724 v.		
45 seconds	0.739 v.	0.707 v.	0.726 v.		
60 seconds	0.740 v.	0.708 v.	0.726 v.		
75 seconds	0.740 v.	0.710 v.	0.728 v.		
90 seconds	0.740 v.	0.740 v. 0.710 v. 0.728 v.			
105 seconds	0.740 v.	0.711 v.	0.730 v.		
120 seconds	0.740 v.	0.711 v.	0.730 v.		
180 seconds	0.740 v.	0.712 v.	0.730 v.		
240 seconds	0.739 v.	0.713 v.	0.730 v.		



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FIGURE	la
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Series No.	8				
Type of sample	Electrolyte - Potato Starch (40 mls/1 gr) Inhibitor - 0.20% Hg in Zn				
Anodic Polariz	Anodic Polarization Current Density 4.33 ma/sq.cm.				
	Reading taken initially at 700F	Reading taken after 3 months at 113° F	Reading taken after 6 months at 1130 F		
O.C.V. before polarization	0.785 v.	0.785 v. 0.777 v. 0.762 v.			
Time on polarization		Anodic Voltage			
1 second	0.768 v.	0.728 v.	. 0.723 v.		
15 seconds	0.769 v.	0.754 v.	0.738 v.		
30 seconds	0.769 v.	0.755 v.	0.740 v.		
45 seconds	0.769 v.	0.756 y. 0.741 y.			
60 seconds	0.768 v.	0.756 v.	0.741 v.		
75 seconds	0.767 ₹.	0.767 v. 0.756 v. 0.742 v.			
90 seconds	0.767 v.	0.767 v. 0.757 v. 0.743 v.			
105 seconds	0.766 v.				
120 seconds	0.766 v.	0.757 v.	0.743 v.		
180 seconds	0.764 v.	0.757 v.	0.745 v.		
240 seconds	0.763 v.	0.758 v.	0.745 v		

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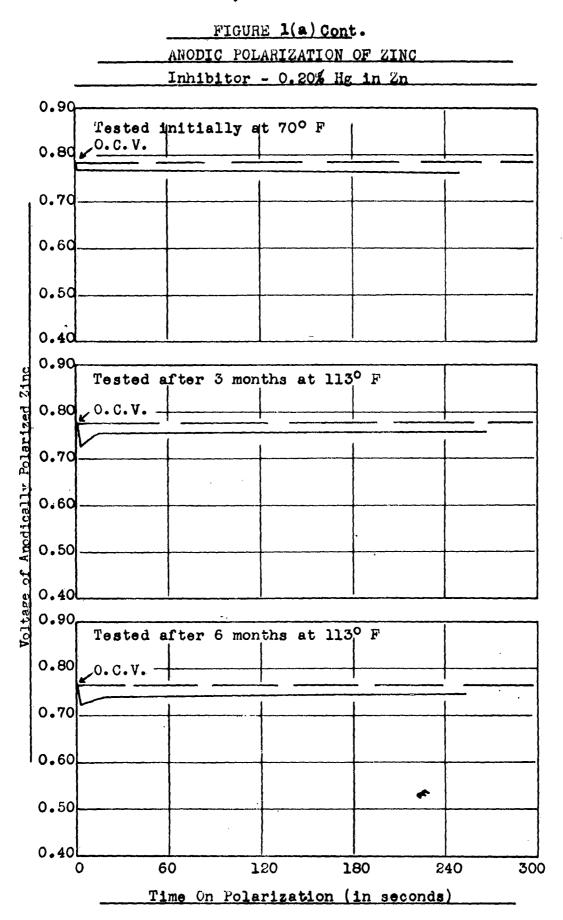
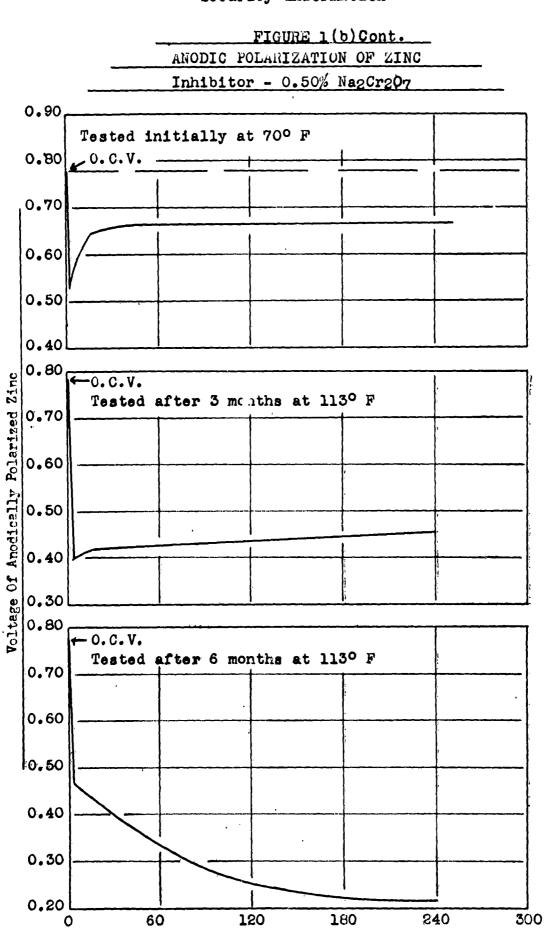


FIGURE 1(b)

Series No.	15	15		
Type of sample	Electrolyte - Pot Inhibitor - 0.50%	Electrolyte - Potato Starch (40 mls/l gr) Inhibitor - 0.50% Sodium Dichromate		
Anodic Polaria	ation Current Dens	ity 4	.33 ma/sq.cm.	
	Reading taken initially at 7 0°F	Reading taken after 3 months at 113° F	Reading taken after 6 months at 1130 P	
O.C.V. before polarization	0.718 v.	0.781 v.	0.774 V.	
Time on polarization		Anodic Voltage		
1 second	0.528 v.	0.398 v.	0.468 v.	
15 seconds	0.648 v.	0.423 v.	0.428 v.	
30 seconds	0.659 v.	0.416 v.	0.397 v.	
45 seconds	0.663 v.	0.424 v.	0.368 v.	
60 seconds	0.664 v.	0.426 v.	0.348 v.	
75 seconds	0.664 v.	0.422 v.	0.298 v.	
90 seconds	0.664 v.	0.428 v.	0.284 v.	
105 seconds	0.665 v.	0.433 v.	0.270 v.	
120 seconds	0.665 v.	0.436 v.	0.253 v.	
180 seconds	0.664 v.	0.448 v.	0.227 v.	
240 seconds	0.664 v.	0.452 v.	0.217 v.	

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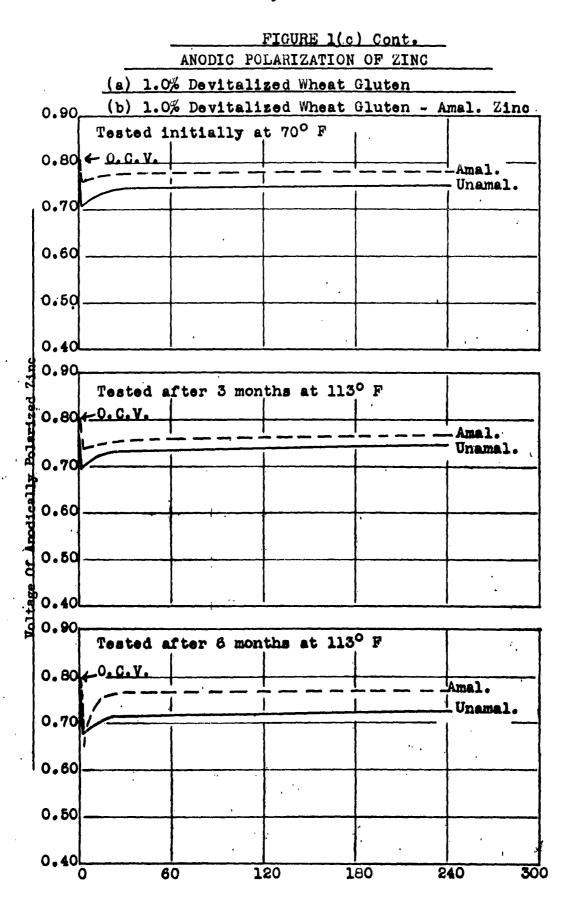
Time On Polarization (in seconds)
Security Information
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FIGURE	1(C

Series No.	3		•		
Type of sample	Electrolyte - Pote Inhibitor - 1.0% W	Electrolyte - Potato Starch (40 mls/l gr) Inhibitor - 1.0% Wheat Gluten (unamal. zinc)			
Anodic Polariza	odic Polarization Current Density 4.33 ma/sq.cm.				
	Reading taken initially at 70° F	Reading taken after 3 months at 1130 F	Reading taken after 6 months at 113° F		
O.C.V. before polarization	0.783 v.	0.775 v.	0.770 v.		
Time on polarization	Anodic Polarization				
1 second	0.708 v.	0.698 v.	0.678 v.		
15 seconds	0.738 v.	0.726 v.	0.709 v.		
30 seconds	0.741 v.	0.730 v.	0.715 v.		
45 seconds	0.744 v.	0.733 v.	0.717 v.		
60 seconds	0.746 v.	0.736 v.	0.719 v.		
75 seconds	0.747 v.	0.737 v.	0.720 v.		
90 seconds	0.748 v.				
105 seconds	0.749 v.	0.739 v.	0.722 v.		
120 seconds	0.749 v.	0.740 v.	0.723 v.		
180 seconds	0.751 v.	0.743 v.	0.724 v.		
240 seconds	0.751 v.	0.745 v.	0.726 v.		

FIGURE 1(c) Cont.

Series No	43		
Type of sample	Electrolyte - Potato Starch (40 mls/l gr) Inhibitor - 1.0% Wheat Gluten and amal. zinc		
Anodic Polariza	ation Current Density 4.33 ma/sq.cm.		
	Reading taken initially at 70° F	Reading taken after 3 months at 113° F	
0.C.V. before polarization	0.801 v.	0.796 v.	0.794 v.
Time on polarization	Anodic Voltage		
l second	0.758 v.	0.738 v.	0.643 v.
15 seconds	0.772 v.	0.750 v.	0.758 v.
30 seconds	0.776 v.	0.754 v.	0.761 v.
45 seconds	0.778 v.	0.757 v.	0.763 v.
60 seconds	0.779 v.	0.759 v.	0.764 v.
75 seconds	0.780 v.	0.760 v.	0.765 v.
90 seconds	0.780 v.	0.761 v.	0.765 v.
105 seconds	0.781 v.	0.762 v.	0.766 v.
120 seconds	0.781 v.	0.763 v.	0.767 v.
180 seconds	0.781 v.	0.765 v.	0.768 v.
240 seconds	0.781 v.	0.766 v.	0.764 v.



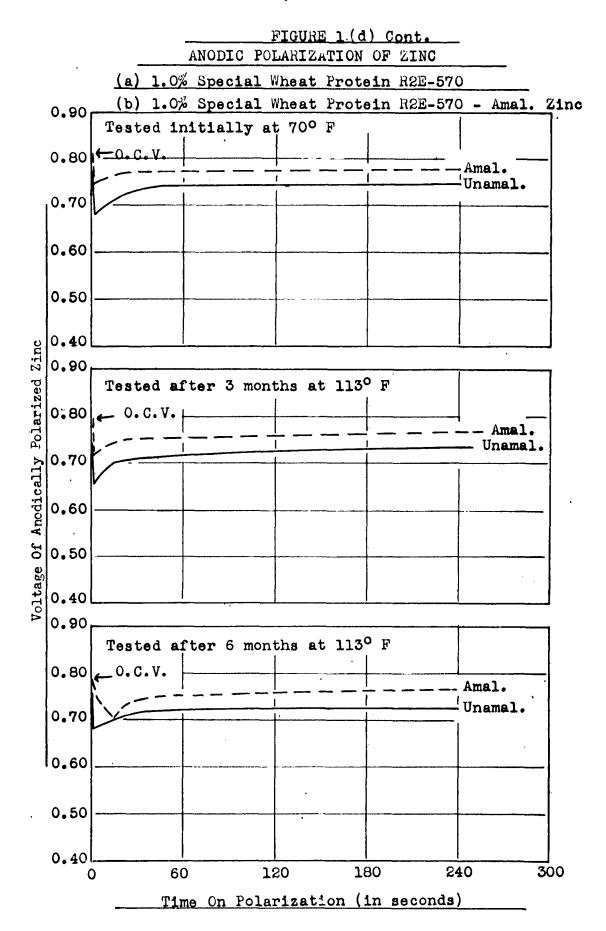
Time On Polarization (in seconds)

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Series No.	4			
Type of sample	Electrolyte - Pote Inhibitor - 1.0%	ato Starch (40 ml Protein R2E-570 (s/l gr) unamal. zinc)	
Anodic Polariza	ation Current Densi	ty 4.	33 ma/sq.cm.	
	Reading taken initially at 70° F	Reading taken after 3 months at 1130 F	Reading taken after 6 months at 113° F	
O.C.V. before polarization	0.781 v.	0.767 v.	0.757 v.	
Time on polarization		Anodic Voltage		
1 second	0.678 v.	0.658 v.	0.681 v.	
15 seconds	0.718 v.	0.701 v.	0.699 v.	
30 seconds	0.736 v.	0.711 v.	0.717 v.	
45 seconds	0.741 v.	0.716 v.	0.719 v.	
60 seconds	0.742 v.	0.719 v.	0.720 v.	
75 seconds	0.744 v.	0.722 v.	0.722 v.	
90 seconds	0.744 v.	0.724 v.	0.723 v.	
105 seconds	0.745 v.	0.7 2 6 v.	0.724 v.	
120 seconds	0.746 v.	0.727 v.	0.725 v.	
180 seconds	0.747 v.	0.731 v.	0.727 v.	
240 seconds	0.748 v.	0.733 v.	0.728 v.	

FIGURE 1(d) Cont.

Series No.	44			
Type of sample	Electrolyte - Potato Starch (40 mls/l gr) Inhibitor - 1.0% Protein R2E570 and amal. zinc			
Anode Polarizati	ion Current Density 4.33 ma/sq.cm.			
	Reading taken initially at 70° F	Reading taken after 3 months at 1130 F	Reading taken after 6 months at 113° F	
0.C.V. before polarization	0.801 v.	0.795 v.	0.784 v.	
Time on polarization	A	Anodic Voltage		
1 second	0.748 v.	0.718 v.	0.754 v.	
15 seconds	0.769 v.	0.748 v.	0.713 v.	
30 seconds	0.771 v.	0.750 v.	0.741 v.	
45 seconds	0.773 v.	0.752 v.	0.747 v.	
60 seconds	0.774 v.	0.754 v.	0.749 v.	
75 seconds	0.774 v.	0.756 v.	0.751 v.	
90 seconds	0.775 v.	0.757 v.	0.754 v.	
105 seconds	0.775 v.	0.758 v.	0.755 v.	
120 seconds	0.775 v.	0.759 v.	0.756 v.	
180 seconds	0.775 v.	0.762 v.	0.766 v.	
240 seconds	0.775 v.	0.764 v.	0.767 v.	



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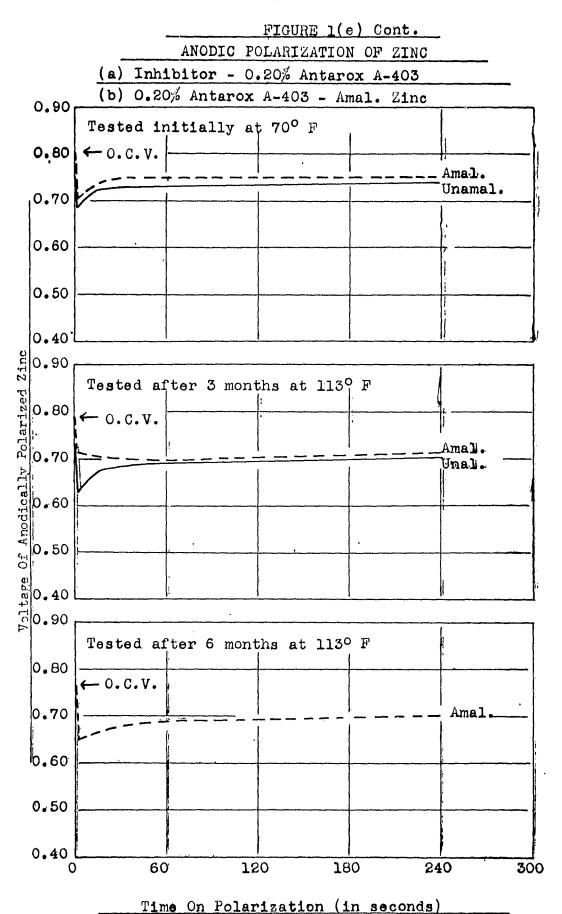
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FIGURE 1(e)

Series No.	28				
Type of sample	Electrolyte - Potato Starch (40 mls/l gr) Inhibitor - 0.20% Antarox A-403 (unamal. zinc)				
Anodic Polarizat	Anodic Polarization Current Density 4.33 ma/sq.cm.				
	Reading taken initially at 70° F	Reading taken after 3 months at 113° F	Reading taken after 6 months at 113° F		
0.C.V. before polarization	0.789 v.	0.762 v.	0.338 v.		
Time on polarization		Anodic Voltage			
1 second	0.688 v.	0.628 v.	0.168 v.		
15 seconds	0.722 v.	0.675 v.	0.068 v.		
30 seconds	0.728 v.	0.684 v.	0.052 v.		
45 seconds	0.732 v.	0.689 v.	0.051 v.		
60 seconds	0.733 v.	0.692 v.	0.052 v.		
75 seconds	0.735 v.	0.693 v.	0.050 v.		
90 seconds	0.736 v.	0.695 v.	0.045 v.		
· 105 seconds	0.736 v.	0.696 v.	0.040 v.		
120 seconds	0.737 v.	0.697 v.	0.036 v.		
180 seconds	0.739 v.	0.700 v.	0.019 v.		
240 seconds	0.739 v.	0.702 v.	0.010 v.		

		/ \ ~	
FIG	FURE 1	(e.)	cont.

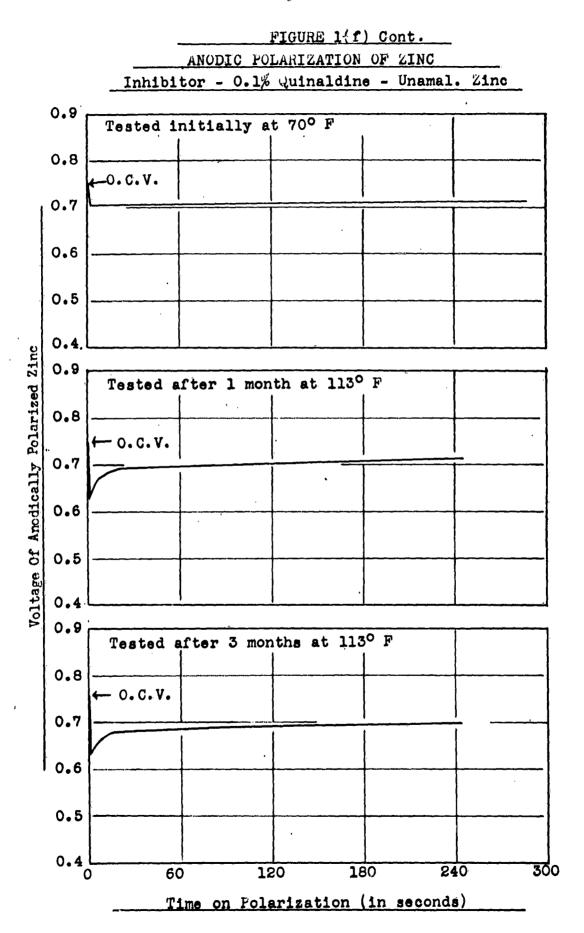
Series No.	33	33								
Type of sample	Electrolyte - Potato Starch (40 mls/l gr) Inhibitor - 0.20% Antarox A-403 and amal. zinc									
Anodic Polariza	tion Current Densi	ty 4.33	ma/sq.cm.							
Reading taken initially at after 3 months after 6 at 113° F. Reading taken after 6 at 113° F.										
0.C.V. before polarization	0.801 v.	0.795 v.	0.768 v.							
Time on polarization	Anodic Voltage									
1 second	0.698 v.	0.718 v.	0.648 v.							
15 seconds	0.739 v.	0.704 v.	0.669 v.							
30 seconds	0.744 v.	0.699 v.	0.678 v.							
45 seconds	0.746 v.	0.697 v.	0.682 v.							
60 seconds	0.747 v.	0.697 v.	0.685 v.							
75 seconds	0.747 v.	0.699 v.	0.687 v.							
90 seconds	0.747 v.	0.701 v.	0.689 v.							
105 seconds	0.747 v.	0.703 v.	0.691 v.							
120 seconds	0.747 v.	0.704 v.	0.692 v.							
180 seconds	0.747 v.	0.711 v.	0,698 v.							
240 seconds	0.747 v.	0.715 v.	0.700 v.							



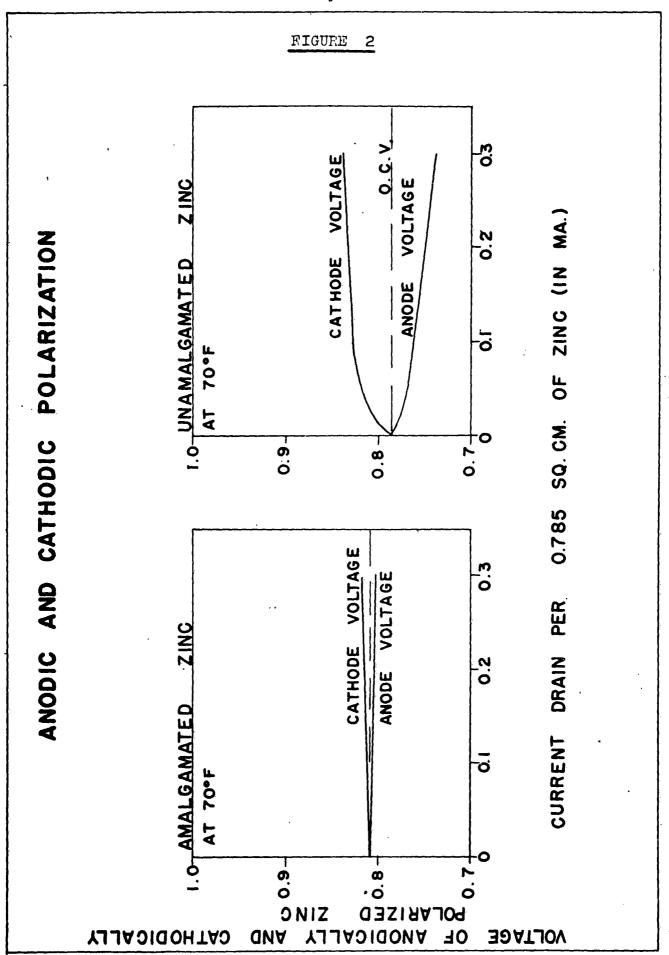
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FIGURE 1(f)

Series No.	47							
Type of sample	Electrolyte - Potato starch (40 ml/l gr.) Inhibitor - 0.1% Quinaldine - unamal. zinc							
Anodic polarization current density 4.33 ma/sq.cm								
	Initial reading at 70° F.	Reading after 3 months storage at 113° F.						
0.C.V. before polarization	0.748 v.	0.749 v.	0.752 v.					
Time on polarization	Anode Voltage							
1 second	0.708 v.	0.638 v.	0.638v.					
15 seconds	0.708 v.	0.690 v.	0.678 v.					
30 seconds	0.709 v.	0.695 v.	0.683 v.					
45 seconds	0.710 v.	0.698 v.	0.686 v.					
60 seconds	0.710 v.	0.7 Ol v.	0.688 v.					
75 seconds	0.710 v.	0.703 v.	0.690 v.					
90 seconds	0.711 v.	0.705 v.	0.691 v.					
105 seconds	0.711 v.	0.706 v.	0.692 v.					
120 seconds	0.711 v.	0.707 v.	0.694 v.					
180 seconds	0.712 v.	0.711 v.	0.697 v.					
240 seconds	0.713 v.	`0.713 v.	0.699 v.					



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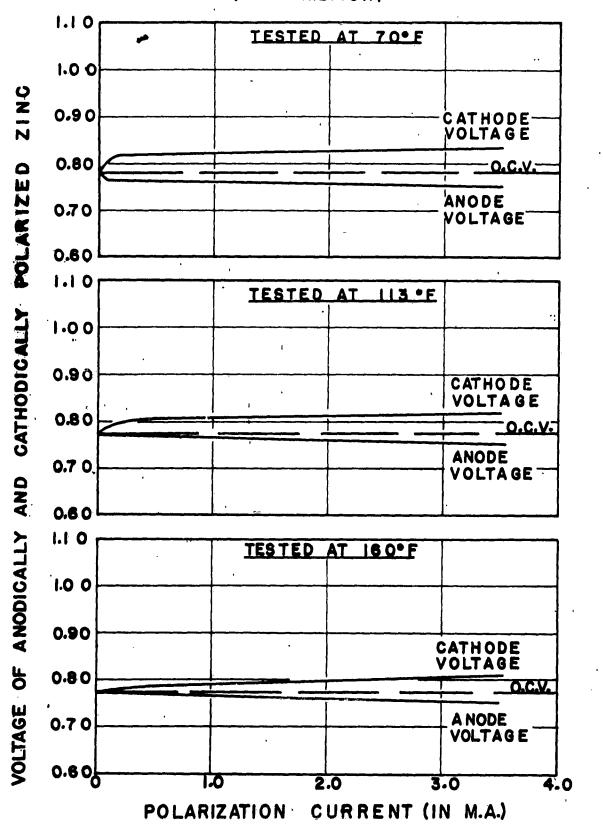
FIGURE 3

	Anodic and	l Cathodic	Polarizati	lon of Zine	<u> </u>	····	
Sample No. A-O							
Type of sample	Electrol no inhib	Electrolyte - Potato Starch (40 mls/1 gm.) with no inhibitor - unamalgamated zinc only.					
Electrolyte Composition		24% NH ₄ Cl; 22.5% ZnCl ₂ ; 53.5% H ₂ O (Composition by weight)					
Area of zinc anode	,	0.785 sq.cm.	Area c	of zinc le	1	785 • cm•	
	Tested @	70°F	Tested @	113°F	Tested @	160 ° F	
	0.C.V.	0.780 v.	0.C.V.	0.776 v.	0.C.V.	0.773 v.	
Polarization Current	Anode Voltage	Cathode Voltage	Anode Voltage	Cathode Voltage	Anode Voltage	Cathode Voltage	
0.08 ma.	0.767 v.	0.810 y.	0.772 v.	0.790 v.	0.772 v.	0.777 v	
0.16 ma.	0.766 v.	0.818 v.	0.769 v.	0.797 v.	0.771 v.	0.779 v	
0.30 ma.	0.766 v.	0.819 v.	0.767 v.	0.801 v.	0.768 v.	0.784 v	
0.50 ma.	0.765 v.	0.821 v.	0.767 v.	0.807 v.	0.767 v.	0.787 v	
1.00 ma.	0.767. v.	0.824 v.	0.764 v.	0.811 v.	0. 764v.	0.793 v	
1.50 ma.	0.759 v.	0.828 v.	0.761 v.	0.812 v.	0.762 v.	0.796 v	
2.00 ma.	0.757 v.	0.828 v.	0.758 v.	0.812 v.	1	1	
2.50 ma.	0.756 v.	0.831 v.	0.756 v.	0.815 v.	0.758 v.	0.802	
3.00 ma.	0.753 v.	0.832 v.	0.755 v.	0.817 v.	0.755 v.	0.805	
3.50 ma.	0.752 v.	0.834 v.	0.753 v.	0.820 v.	0.753 v.	0.807	

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FIGURE 3

ANODIC AND CATHODIC POLARIZATION OF ZINC SAMPLE A-O (NO INHIBITOR)



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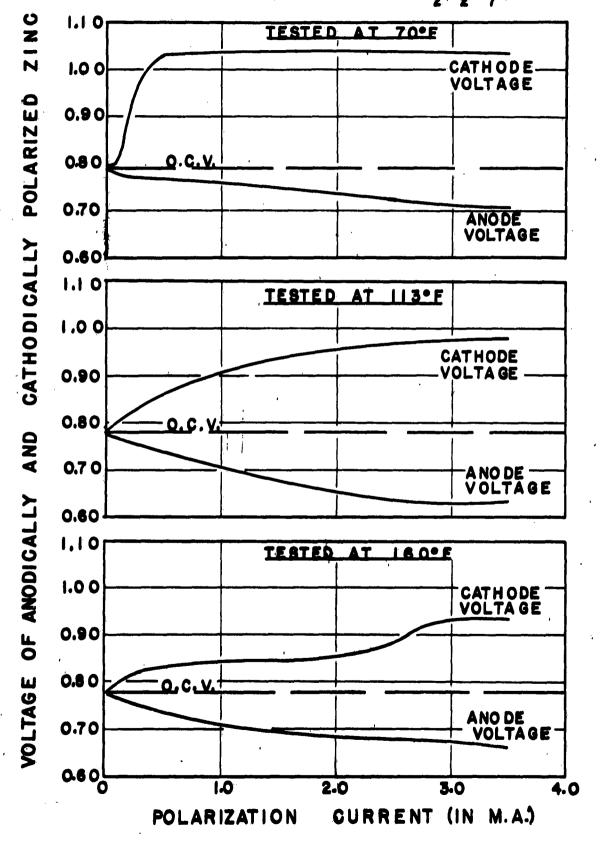
FIGURE	3(a)

	Anodic (and (Cathodic	Pc	larizat	ion of	Zin	C		
Sample No.								A-	<u> 1</u>	
Type of sample	Electro 0.20%	Electrolyte - Potato Starch (40 mls/l gm.) with 0.20% Na ₂ Cr ₂ O ₇ as inhibitor								
Electrolyte Composition					; 22.5%			53.5%	H ₂ 0	
Area of zinc anode			.785 q.cm.		Area o					.785 q.cm.
	Tested	Tested at 70°F Tested at 113°F Tes				Test	ed at	160°F		
	0.C.V.	0.	.793 v .	0.	C.V.	0.780	٧.	0.C.	٧.	0.778 v.
Polarization Current	Anode Voltage		athode oltage	_	node ltage	Catho Volta		Ano Volt		Cathode Voltage
0.08 ma.	0.779 v	. 0	.797 v.	0.	768 v.	0.800	v.	0.77	5 v.	0.787 v.
0.16 ma.	0.772 v	. 0.	.839 v.	0.	761 v.	0.812	٧.	0.76	4 v.	0.807 v.
0.30 ma.	0.772 v	• O	.987 v.	0.	750 v.	0.835	v.	0.74	9 v.	0.822 v.
0.50 ma.	0.768 v	. 0	.999 v.	0.	732 v.	0.847	v.	0.73	9 v.	0.827 v.
1.00 ma.	0.761 v	. 1.	.032 v.	0.	709 v.	0.906	٧.	0.71	2 v.	0.839 v.
1.50 ma.	0.744 v	. 1.	.033 v.	0.	672 v.	0.958	v.	0.69	7 v.	0.844 v.
2.00 ma.	0.735 v	. 1	.033 v.	0.	651 v.	0,955	v.	0.68	8 v.	0.853 v.
2.50 ma.	0.725 v	. 1.	.031 v.	0.	639 v.	0.958	v •	0.67	6 V.	0.883 v.
3.00 ma.	0.717 v	. 1	.035 v.	0.	63 3 v.	0.963	v.	0.67	3 v.	0.933 v .
3.50 ma.	0.709 v	. 1	.034 v.	0.	635 v.	0.988	v.	0.66	3 v.	0.931 v.

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FIGURE 3A

ANODIC AND CATHODIC POLARIZATION OF ZINC SAMPLE A-I (INHIBITOR - .20% NA2CR2 07)



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FIGURE	3(b)

						·———			
Anodic and Cathodic Polarization of Zinc									
Sample No.					A-2	:-			
Type of sample		Electrolyte - Potato Starch (40 mls/1 gm.) with 0.25% Mercury in Zinc as inhibitor.							
Electrolyte Composition		24% NH4Cl; 22.5% ZnCl2; 53.5% H2O (Composition by weight)							
Area of zinc anode		0.785 Area of zinc sq.cm. cathode							
	Tested	at 70°F	Tested at	113°F	Tested at	: 160 ⁰ F			
	0.C.V.	0.790 v.	0.C.V.	0.785 v.	0.C.V.	0.776 v.			
Polarization Current	Anode Voltage	Cathode Voltage	Anode Voltage	Cathode Voltage	Anode Voltage	Cathode Voltage			
0.08 ma.	0.785 v.	0.791 v.	0.785 v.	0.786 v.	0.776 v.	0.776 v.			
0.16 ma.	0.781 v.	0.794 v.	0.784 v.	0.788 v.	0.776 v.	0.776 v.			
0.30 ma.	0.775 v.	0.798 v.	0.783 v.	0.791 v.	0.775 v.	0.777 v.			
0.50 ma.	0.767 v.	0.804 v.	0.782 v.	0.797 v.	0.775 v.	0.778 v.			
1.00 ma.	0.756 v.	0.818 v.	0.780 v.	0.808 v.	0.773 v.	0.779 v.			
1.50 ma.	0.750 v.	0.831 v.	0.778 v.	0.816 v.	0.772 v.	0.781 v.			
2.00 ma.	0.746 v.	0.828 v.	0.776 v.	0.818 v.	0.771 v.	0.782 v.			
2.50 ma.	0.752 v.	0.829 v.	0.775 v.	0.818 v.	0.770 v.	0.784 v.			
3.00 ma.	0.750 v.	0.827 v.	0.772 v.	0.819 v.	0.770 v.	0.786 v.			
3.50 ma.	0.752 v.	0.829 v.	0.770 v.	0.821 v.	0.769 v.	0.788 v.			

FIGURE 38

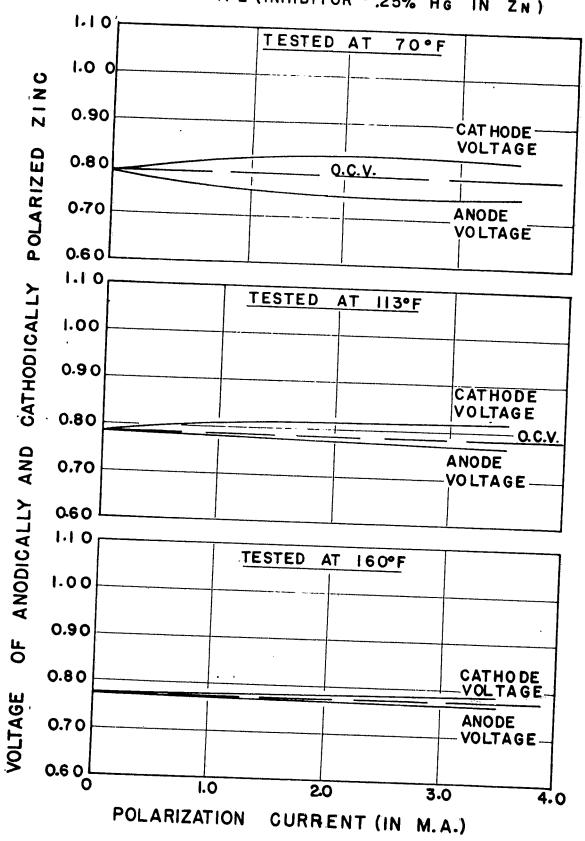
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ANODIC AND CATHODIC POLARIZATION OF ZII SAMPLE A-2 (INHIBITOR - .25% Hg IN ZN)



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	FIGURE	3(c)	
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Anodic and Cathodic Polarization of Zinc									
Sample No. A-3									
Type of sample		Electrolyte - Potato Starch (40 mls/1 gm.) with 0.20% Antaron R-155 as inhibitor							
Electrolyte Composition	,	24% NH4CL; 22.5% ZnCl2; 53.5% H2O (Composition by weight)							
Area of zinc		0.785 sq.cm.	Area catho	of zinc de	ı	.785 q.cm.			
	Tested a	t 70°F	Tested a	t 113 ⁰ F	Tested at	160 ° F			
•	0.C.V.	0.771 v.	0.C.V.	0.773 v.	0.C.V.	0.753 v.			
Polarization Current	Anode Voltage	Cathode Voltage	Anode Voltage	Cathode Voltage	Anode Voltage	Cathode Voltage			
0.08 ma.	0.749 v.	0.867 v.	0.715 v.	0.825 v.	0.730 v.	0.808 v.			
0.16 ma.	0.731 v.	0.874 v.	0.712 v.	0.848 v.	0.715 v.	0.829 v.			
0.30 ma.	0.730 v.	0.890 v.	0.709 v.	0.877 v.	0.693 v.	0.850 v.			
0.50 ma.	0.720 v.	0.887 v.	0.708 v.	0.886 v.	0.677 v.	0.869 v.			
1.00 ma.	0.716 v.	0.884 v.	0.707 v.	0.879 v.	0.679 v.	0.881 v.			
1.50 ma.	0.717 v.	0.887 v.	0.709 v.	0.879 v.	0.669 v.	0.874 v.			
2.00 ma.	0.718 v.	0.890 v.	0.707 v.	0.881 v.	0.669 v.	0.876 v.			
2.50 ma.	0.716 v.	0.894 v.	0.708 v.	0.881 v.	0.662 v.	0.876 v.			
3.00 ma.	0.714 v.	0.897 v.	0.707 v.	0.884 v.	0.670 v.	0.876 v.			
3.50 ma.	0.712 v.	0.900 v.	0.704 v.	0.887 v.	0.672 v.	0.875 v.			

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FIGURE 3c

ANODIC AND CATHODIC POLARIZATION OF ZINC SAMPLE A-3 (INHIBITOR - .20% ANTARON R-155)

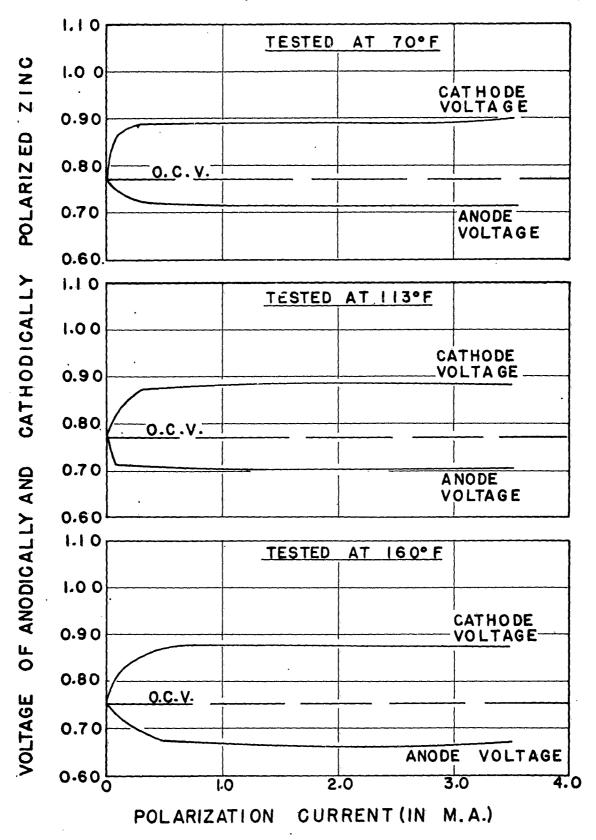
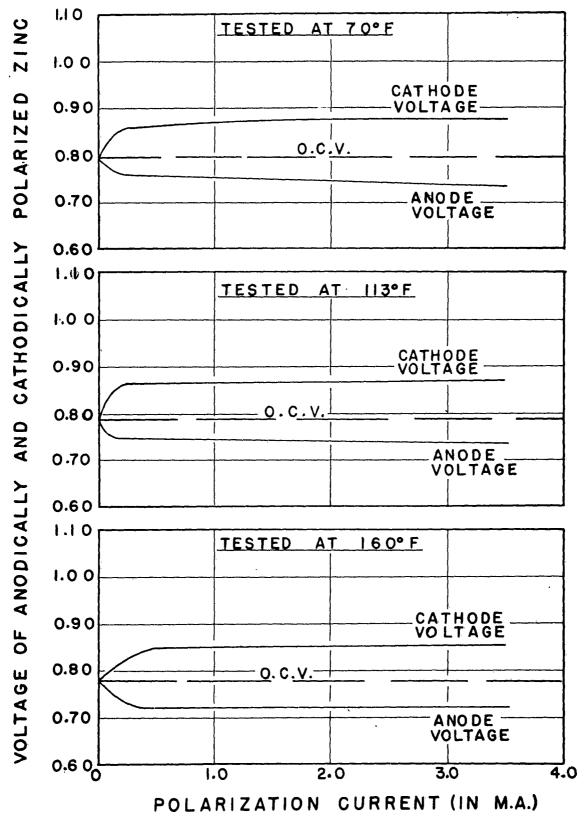


FIGURE 3(d)

<u></u>							
Anodic and Cathodic Polarization of Zinc							
Sample No.			**************************************		A-4		
Type of sample		Electrolyte - Potato Starch (40 mls/l gm.) with 0.20% Antarox A-403 as inhibitor					
Electrolyte Composition		24% NH4Cl; 22.5% ZnCl2; 53.5% H2O (Composition by weight)					
Area of zinc anode		0.785 sq.cm.	Area o	of zinc de	•	785 [•cm•	
	Tested a	t 70°F	Tested a	t 113 ⁰ F	Tested at	: 160 ⁰ F	
	0.C.V.	0.794 v.	0.C.V.	0.788 v.	0.C.V.	0.779 v.	
Polarization Current	Anode Voltage	Cathode Vollage	Anode Voltage	Cathode Voltage	Anode Voltage	Cathode Voltage	
0.08 ma.	0.777 v.	0.821 v.	0.754 v.	0.833 v.	0.756 v.	0.790 v.	
0.16 ma.	0.764 v.	0.861 v.	0.748 v.	0.855 v.	0.740 v.	0.813 v.	
0.30 ma.	0.761 v.	0.860 v.	0.748 v.	0.864 v.	0.726 v.	0.829 v.	
0.50 ma.	0.759 v.	0.863 v.	0.743 v.	0.863 v.	0.720 v.	0.846 v.	
1.00 ma.	0.756 v.	0.865 v.	0.743 v.	0.859 v.	0.725 v.	0.859 v.	
1.50 ma.	0.750 v.	0.868 v.	0.742 v.	0.863 v.	0.722 v.	0.847 v.	
2.00 ma.	0.747 v.	0.872 v.	0.739 v.	0.863 v.	0.728 v.	0.850 v.	
2.50 ma.	0.744 v.	0.873 v.	0.738 v.	0.866 v.	0.724 v.	0.849 v.	
3.00 ma.	0.740 v.	0.877 v.	0.736 v.	0.867 v.	0.725 v.	0.851 v.	
3.50 ma.	0.737 v.	0.879 v.	0.735 v.	0.870 v.	0.723 v.	0.851 v.	

FIGURE 30
ANODIC AND CATHODIC POLARIZATION OF ZINC
SAMPLE A-4 (INHIBITOR - 20% ANTAROX A-403)



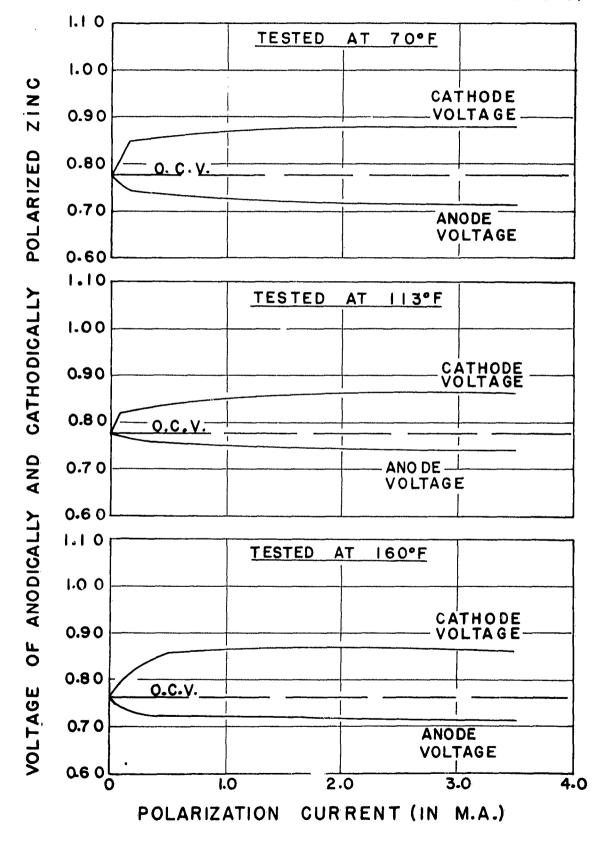
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FIGURA	3(e)

f 				·			
	Anodic and	i Cathodic	Polarizati	on of Zinc	;		
Sample No.					A-5	<u> </u>	
Type of sample	Electrolyte - Potato Starch (40 mls/l gm.) with 1.0% Protein R2E-570 as inhibitor						
Electrolyte Composition.							
Area of zinc		0.785 Area of zinc 0.785 sq.cm. cathode sq.cm.				-	
	Tested a	at 70°F	Tested at 113°F T			ested at 160°F	
	0.C.V.	0.777 v.	0.C.V.	0.777 v.	0.C.	v.	0.766 v.
Polarization Current	Anode Voltage	Cathode Voltage	Anode Voltage	Cathode Voltage	And Volt		Cathode Voltage
0.08 ma.	0.752 v.	0.807 v.	0.772 v.	0.820 v.	0.74	0 v.	0.7998
0.16 ma.	0.745 v.	0.847 v.	0.766 v.	0.825 v.	0.72	27 v.	0.815 v.
0.30 ma.	0.745 v.	0.855 v.	0.760 v.	0.833 v.	0.73	0 v.	0.842 v.
0.50 ma.	0.730 v.	0.861 v.	0.756 v.	0.839 v.	0.72	4 v.	0.860 v.
i.00 ma.	0.730 v.	0.867 v.	0.751 v.	0.849 v.	0.72	22 v.	0.863 v.
1.50 ma.	0.723 v.	0.873 v.	0.749 v.	0.854 v.	0.71	2 v.	0.862 v.
2.00 ma.	0.721 v.	0.877 v.	0.746 v.	0.856 v.	0.73	4 v.	0.863 v.
2.50 ma.	0.717 v.	0.878 v.	0.744 v.	0.860 v.	0.7	4 v.	0.858 v.
3.00 ma.	0.715 v.	0.882 v.	0.741 v.	0.863 v.	0.7	.5 v.	0.857 v.
3.50 ma.	0.712 v.	0.881 v.	0.739 v.	0.866 v.	0.7	ll v.	0.855 v.

FIGURE 3E

ANODIC AND CATHODIC POLARIZATION OF ZINC SAMPLE A-5 (INHIBITOR + 1.0% PROTEIN R2E-570)



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FIGURE 3(f)
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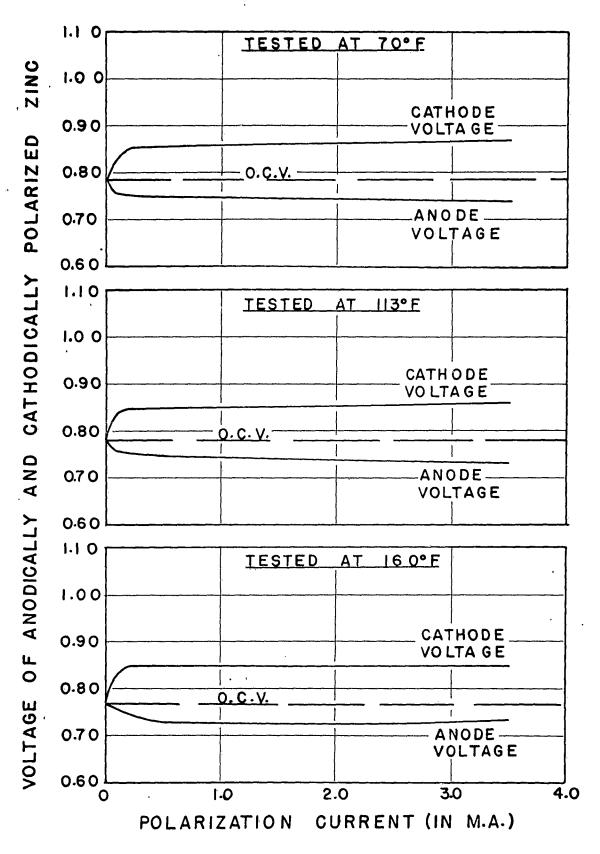
	Anodic a	nd Cathodic	Polarizat	ion of Zin	ıc		
Sample No.	,				A-6		
Type of sample	Electrolyte - Potato Starch (40 mls/1 gm.) with 1.0% Denatured Gluten as inhibitor						
Electrolyte Composition		24% NH4Cl; 22.5% ZnCl2; 53.5% H2O (Composition by weight)					
Area of zinc anode		0.785 sq.cm.					785 .cm.
	Tested	at 7.00F	OF Tested at 113°F			Tested at 160°F	
	0.C.V.	0.784 v.	0.C.V.	0.780 v.	0.C.V.		0.768 v.
Polarization Current	Anode Voltage	Cathode Voltage	Anode Voltage	Cathode Voltage	Anode Voltage	•	Cathode . Voltage
0.08 ma.	0.759 v	0.807 v.	0.758 v.	0.841 v.	0.757	v.	0.826 v.
0.16 ma.	0.755 v	0.853 v.	0.754 v.	0.846 v.	0.749	٧.	0.845 v.
0.30 ma.	0.754 v	. 0.856 v.	0.753 v.	0.847 v.	0.740	v.	0.850 v.
0.50 ma.	0.750 v	0.857 v.	0.745 v.	0.847 v.	0.729	v.	0.844 v.
1.00 ma.	0.747 v	. 0.859 v.	0.746 v.	0.849 v.	0.729	v.	0.849 v.
1.50 ma.	0.746 v	. 0.862 v.	0.743 v.	0.853 v.	0.728	v.	0.845 v.
2.00 ma.	0.743 v	0.863 v.	0.739 v.	0.854 v.	0.737	٧.	0.848 v.
2.50 ma.	0.742 v	. 0.866 v.	0.738 v.	0.858 v.	0.732	v.	0.846 v.
3.00 ma.	0.740 v	0.866 v.	0.735 v.	0.858 v.	0.736	v.	0.847 v.
3.50 ma.	0.739 v	. 0.869 v.	0.734 v.	0.861 v.	0.734	v.	0.847 v.

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FIGURE 3F
ANODIC AND CATHODIC POLARIZATION OF ZINC
SAMPLE A-6 (INHIBITOR - 10% DENATURED GLUTEN)



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FIGURE	3(g)

•	Anodic an	d Cathodic	Polarizat	ion of Zin	ıc		
Sample No.					A-9		
Type of sample	Electrol Hg in zi	Electrolyte - Potato Starch (40 mls/l gm.) with 0.20% Hg in zinc plus 0.20% antaron R-155 as inhibitors					
Electrolyte Composition		24% NH4Cl; 22.5% ZnCl2; 53.5% H2O (Composition by weight)					
Area of zinc anode		0.783 sq.cm.	Area o	f zinc	1	785 .cm.	
	Tested a	t 70°F	Tested a	t 113°F	Tested at	160°F	
	0.C.V.	0.797 v.	0.C.V.	0.789 v.	0.C.V.	0.774 v.	
Polarization Current	Anode Voltage	Cathode Voltage	Anode Voltage	Cathode Voltage	Anode Voltage	Cathode Voltage	
0.08 ma.	0.791 v.	0.821 v.	0.773 v.	0.797 v.	0.765 v.	0.774 v.	
0.16 ma.	0.758 v.	0.841 v.	0.762 v.	0.804 v.	0.762 v.	0.797 v.	
0.30 ma.	0.743 v.	0.866 v.	0.743 v.	0.826 v.	0.754 v.	0.813 v.	
0.50 ma.	0.737 v.	0.890 v.	0.727 v.	0.850 v.	0.744 v.	0.833 v.	
1.00 ma.	0 ,06 v.	0.901 v.	0.702 v.	0.883 v.	0.725 v.	0.859 v.	
1.50 ma.	0.693 v.	0.897 v.	0.686 v.	0.884 v.	0,715 v.	0.876 v.	
2.00 ma.	0.683 v.	0.897 v.	0.674 v.	0.887 v.	0.706 v.	0.878 v.	
2.50 ma.	0.674 v.	0.900 v.	0.665 v.	0.888 v.	0.700 v.	0.886 v.	
3.00 ma.	0.666 v.	0.902 v.	0.657 v.	0.889 v.	0.693 v.	0.886 v.	
3.50 ma.	0.659 v.	0.904 v.	0.650 v.	0.892 v.	0.689 v.	0.888 v.	

FIGURE 36 ANODIC AND CATHODIC POLARIZATION ZINC OF SAMPLE A-9 (INHIBITOR - 20% HG IN ZN + .20% ANTARON R-155 1.10 TESTED AT 70°F 1.00 ZINC CATHODE **VOLT AGE** 0.90 CATHODICALLY POLARIZED O.C.V. 0.80 0.70 VOLTAGE ANODE 0.60 1.10 TESTED AT 113°F 1.0 0 CATHODE VOLTAGE 0.90 O.C.V. 0.80 AND ANO DE 0.70 VOLTAGE VOLTAGE OF ANODICALLY 0.60 1.10 TESTED **AT** 160°F 1.00 CATHODE VOLTA GE 0.90 0.80 O.C. V 0.70 ANODE VOLTAGE 0.60 2.0 4.0 3.0 1.0

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POLARIZ ATION

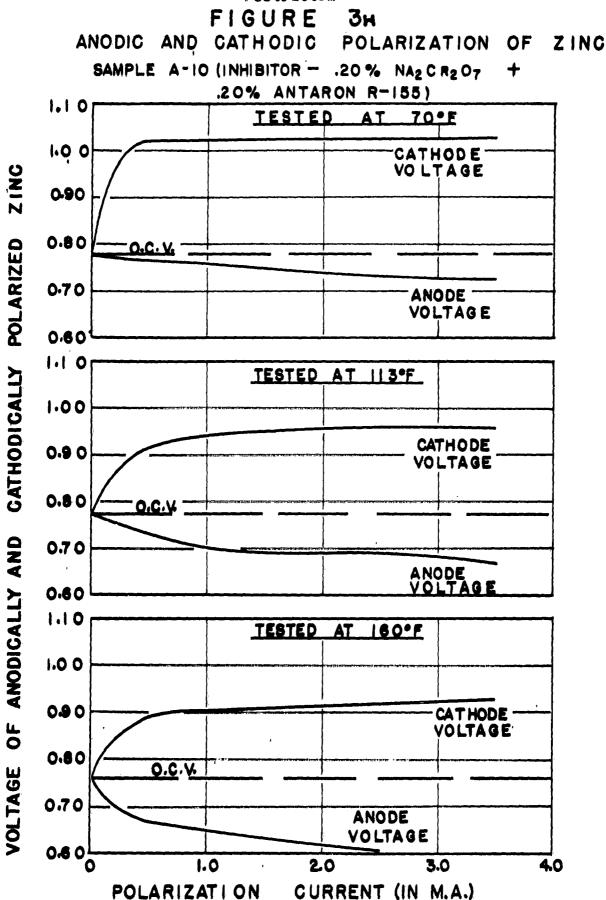
FIGURE 3(h)

	Anodic er	nd Cathodic	Polonizat	iton of 74n		·	
Sample No.	MIOUIC A	id Oadhodic	TOTALIZA	1011 01 211	A-10		
Type of sample	Electro	lyte - Pota plus 0.20	to Starch % Antaron	(40 mls/1 R-155 as i	gm.) nhibi	with tors	0.20%
Electrolyte Composition			401; 22.5 sition by	% ZnCl2; weight)	53.5%	H20	
Area of zinc anode		0.785 sq.cm.	Area c	of zinc le).785 sq.cm.
	Tested a	at 70°F	Tested a	t 113 ° F	Tes	ted a	t 160°F
	O.C.V.	0.780 v.	0.C.V.	0.775 v.	0.C.	v.	0.761 v.
Polarization Current	Anode Voltage	Cathode Voltage	Anode Voltage	Cathode Voltage	Ano Volt		Cathode Voltage
0.08 ma.	0.775 v.	0.880 v.	0.768 v.	0.821 v.	0.72	3 v.	0.802 v.
0.16 ma.	0.774 v.	0.927 v.	0.763 v.	0.855 v.	0.70	9 v.	0.832 v.
0.30 ma.	0.768 v.	0.999 v.	0.744 v.	0.882 v.	0.69	2 v.	0.864 v.
0.50 ma.	0.767 v.	1.025 v.	0.736 v.	0.920 v.	0.66	6 v.	0.892 v.
1.00 ma.	0.759 v.	1.018 v.	0.701 v.	0.947 v.	0.64	8 v.	0.902 v.
1.50 ma.	0.748 v.	1.027 v.	0.689 v.	0.946 v.	0.62	8 v.	0.910 v.
2.00 ma.	0.739 v.	1.032 v.	0.692 v.	0.950 v.	0.61	7 v.	0.919 v.
2.50 ma.	0.732 v.	1.035 v.	0.688 v.	0.953 v.	0.60	3 v.	0.924 v.
3.00 ma.	0.730 v.	1.037 v.	0.679 v.	0.955 v.	0.59	6 v.	0.926 v.
3.50 ma.	0.725 v.	1.036 v.	0.670 v.	0.960 v.	0.58	9 v.	0.929 v.

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FIGURE	3(1)	

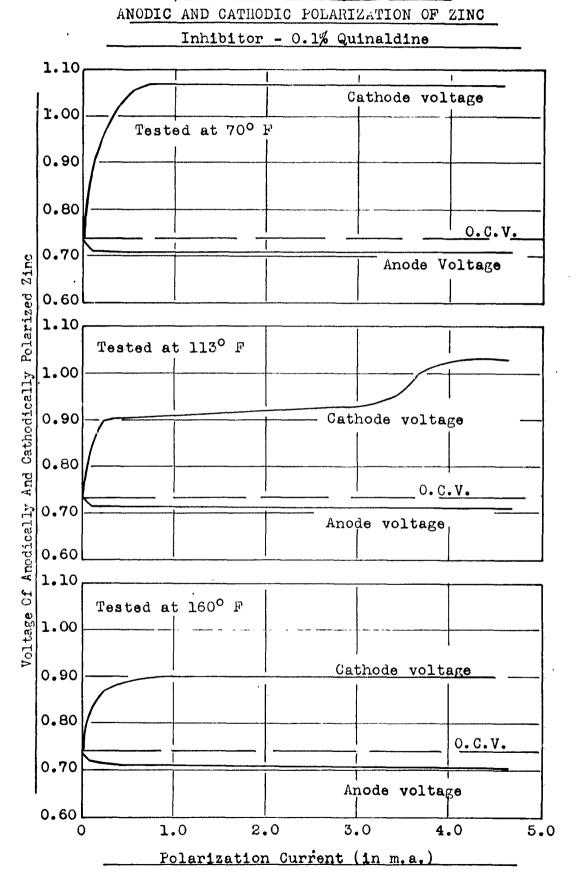
Anode and Cathodic Polarization of Zinc

Sample No.				A	- 13	
Type of sample	Electrol Inhibito	yte - Pota r - 0.1% Q	to Starch uinaldine	(40 mls/l	gm)	
Electrolyte Composition			NH4Cl; 22 sition by	.5% ZnCl2 weight)	; 53.5% H ₂	Ф .
Area of Zinc Anode	3	.785 q.cm.	Area o Cathod		0.78 sq.c	
	Tested a	t 7 0°F	Tested a	t 113°F	Tested a	t 160°F
	0.C.V.	0.736 v.	0.C.V.	0.734 v.	0.C.V.	0.741 v.
Polarization Current	Anode Voltage	Cathode Voltage	Anode Voltage	Cathode Voltage	Anode Voltage	Cathode Voltage
0.08 ma.	0 .14 v.	0.907 v.	0.715 v.	0.858 v.	0.711 v.	0.842 v.
0.16 ma.	0.716 v.	1	0.724 v.	0.894 v.	0.728 v.	0.864 v.
0.30 ma.	0.713 v.		0.714 v.	0.901 v.	0.706 v.	0.884 v.
0.50 ma.	0.714 v.	1.058 v.	0.715 v.	0.904 v.	0.711 v.	0.898 v.
1.00 ma.	0.711 v.	1.068 v.	0.710 v.	0.913 v.	0.700 v.	0.885 v.
1.50 ma.	0.712 v.	1.078 v.	0.711v.	0.922 v.	0.707 v.	0.898 v.
2.00 ma.	0.709 v.	1.073 v.	0.709 v.	0.926 v.	0.704 v.	0.891 v.
2.50 ma.	0.713 v.	1.064 v.	0.709 v.	0.931 v.	0.7 04 v	.0.895 v.
3.00 ma.	0.711 v.	.1.058 v.	0.710 v.	1.012 v.	0.701 v.	0.895 v.
3.50 ma.	0.710 v.	1.068 v.	0.710 v.	1.032 v.	0.702 v.	0.900 v.

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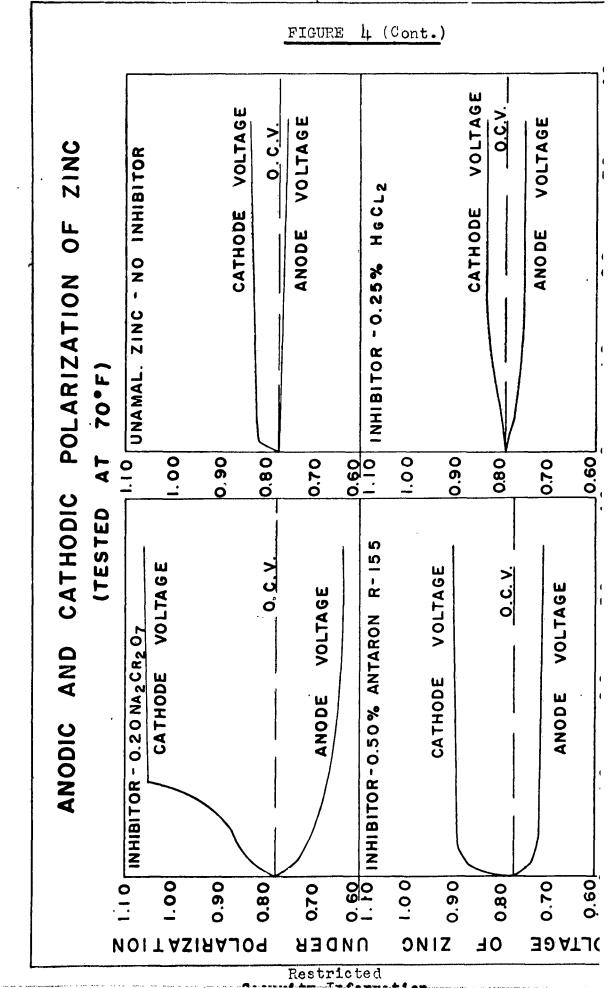
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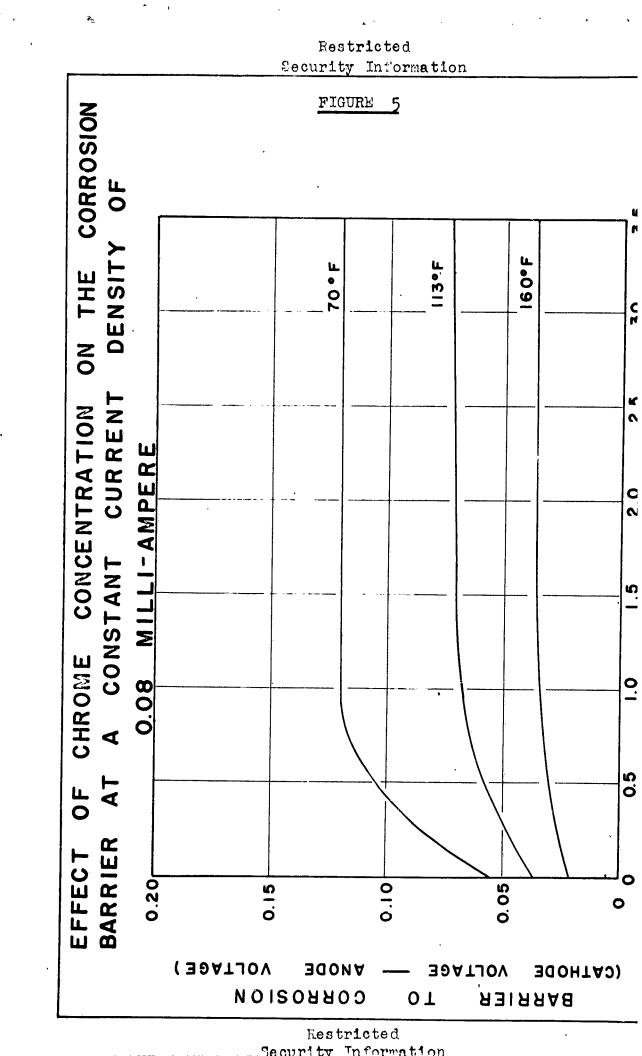
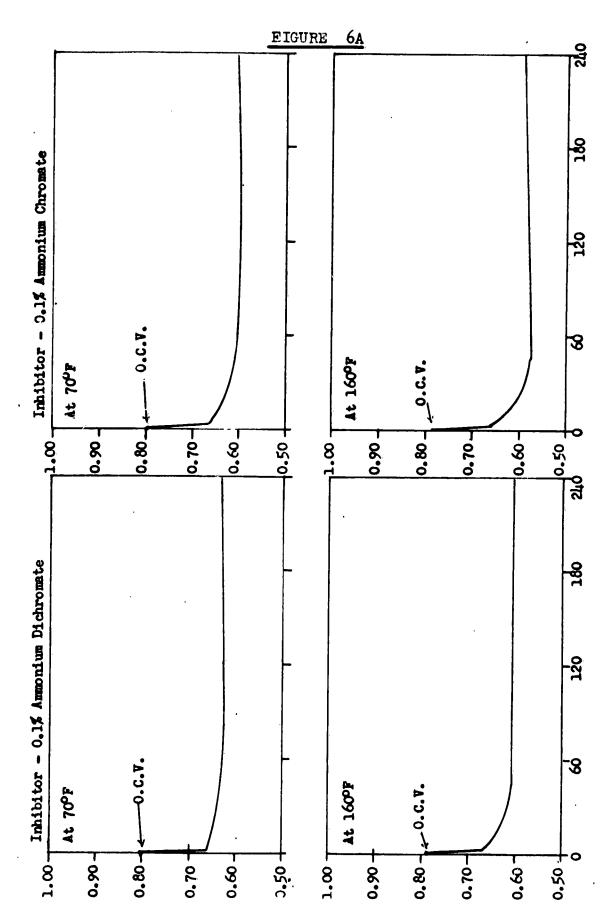


FIGURE 6

	ANODIC PO	DLARIZATION					
Anodic Polarizat	ion Current De	nsity	4.33 ma./sq.	cm.			
Inhibitor	0.1% (NH4)20	Cr207	0.1% (NH4)2Cr	04			
		After 24 hrs at 160°F	After 24 hrs at 70°F	After 24 hrs at 160°F			
0.C.V. before polarization	0.798 v.	0.784 v.	0.795 v.	0.785 v.			
Time polarized		Anodic Voltage					
1 second	0.658 v.	0.668 v.	0.663 v.	0.658 v.			
15 seconds:	0.653 v.	0.632 v.	0.645 v.	0.623 v.			
30 seconds	0.634 v.	0.616 v.	0.623 v.	0.587 v.			
60 seconds	0.626 v.	0.606 v.	0.603 v.	0.577 v.			
90 seconds	0.624 v.	0.603 v.	0.595 v.	0.579 v.			
120 seconds	0.628 v.	0.604 v.	0.595 v.	0.582 v.			
180 seconds	0.630 v.	0.606 v.	0.598 v.	0.589 v.			
240 seconds	0.632 v.	0.608 v.	0.603 v.	0.591 v.			

	. (CATHODIC	AND AND	DIC POL	ARIZATIO	N		
Inhibitor	nhibitor 0.1% (NH4)2Cr2O7					NH4)2Cr	04	
	Tested	@ 70 ⁰ F	Tested	@ 160°F	Tested	@ 70°F	Tested	@ 160°F
	OCV - C	.796 v.	ocv - c	.778 v.	OCV - C	.795 v.	OCV - 0.786 v.	
Polarization Current	Anode Voltage	Cathode Voltage	Anode Voltage	Cathode Voltage		Cathode Voltage		Cathode Voltage
0.01 ma.	0.789	0.802	0.776	0,781	0.791	0.814	0.784	0.790
0.029 ma.	0.780	0.814	0.770	0.784	0.786	0.832	0.780	0.797
0.035 ma.	0.779	0.821	0.768	0.787	0.786	0.840	0.779	0.804
0.046 ma.	0,777	0.837	0.760	0.789	0.786	0.845	0.776	0.809
0.075 ma.	0.772	0.838	0.750	0.798	0.782	0.852	0.772	0.822
0.100 ma.	0.769	0.844	0.739	0.802	0.782	0.857	0.765	0.829
0.150 ma.	0.764	0.850	0.728	0.817	0.781	0.863	0.755	0.843
0.300 ma.	0.750	0.862	0.703	0.830	0.778	0.878	0.736	0.854

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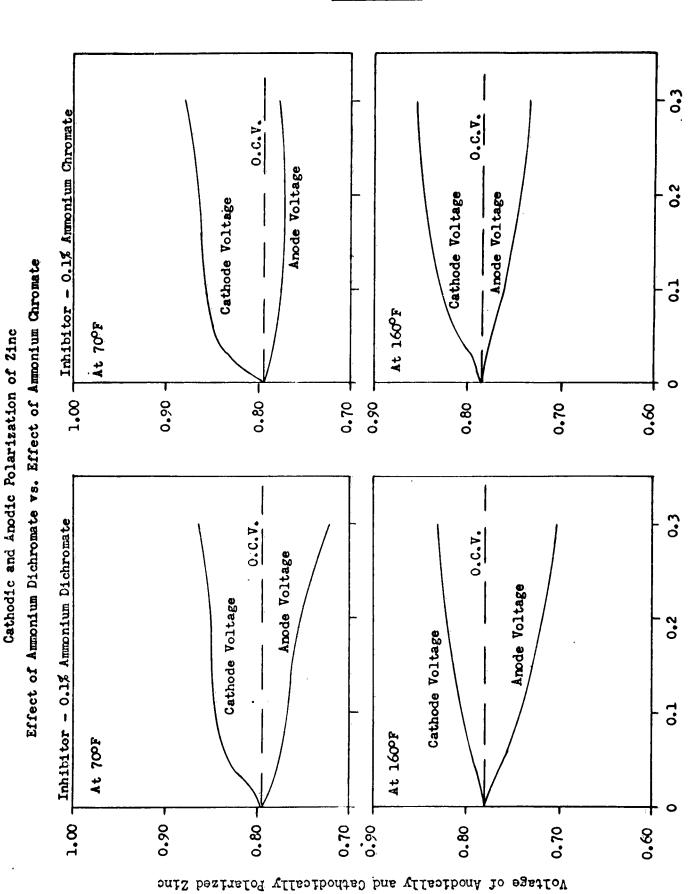
Anodic Polarisation Of Zinc

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Voltage of Anodically Polarized Zinc

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FIGURE 6B



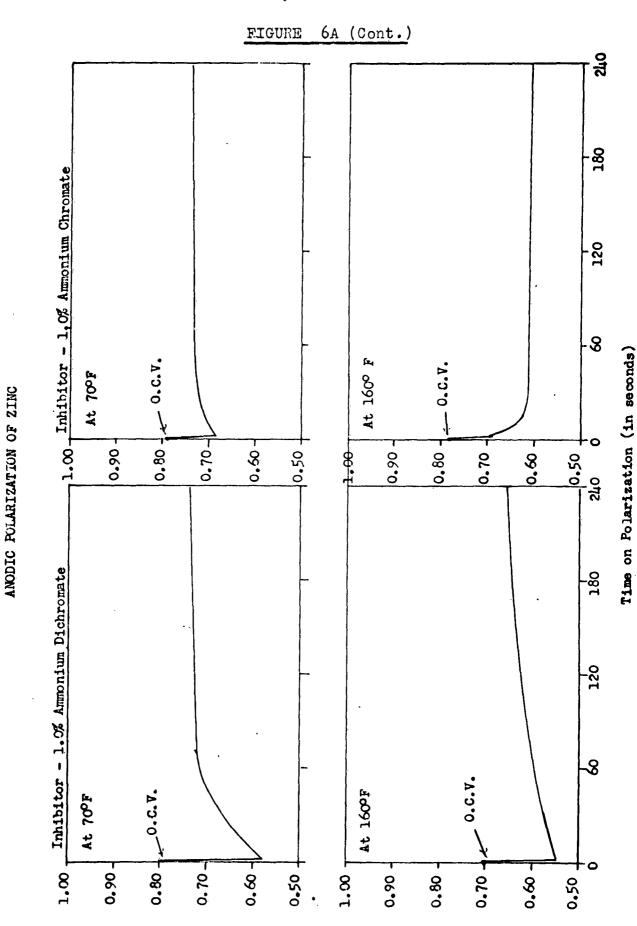
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FIGURE 6 (Cont.)

	ANODI	C POLARIZATIO	N		
Anodic polarizat	ion current de	nsity	4.33 ma./sq.	em.	
Inhibitor	1.0% (NH4)2	Cr ₂ O ₇	1.0% (NH4)2CrC	04	
		After 24 hrs at 160°F	After 24 hrs. at 7 0°F	After 24 hrs. at 160°F	
O.C.V. before polarization	0.789 v.	0.694 v.	0.793 ∀.	0.789 ▼.	
Time polarized					
1 second	0.578 v.	0.548 v.	0.683 v.	0.688 v.	
15 "	0.621 v.	0.556 v.	0.714 v.	0.623 v.	
30 "	0.668 v.	0.576 v.	0.724 v.	0.614 v.	
60_"	0.714 v.	0.597 v.	0.732 v.	0.617 v.	
90 "	0.724 v.	0.611 v.	0.734 v.	0.618 v.	
120 "	0.729 v.	0.621 v.	0.737 v.	0.617 v.	
180 "	0.733 v.	0.640 v.	0.739 v.	0.611 v.	
240 "	0.736 v.	0.657 v.	0.739 v.	0.605 v.	

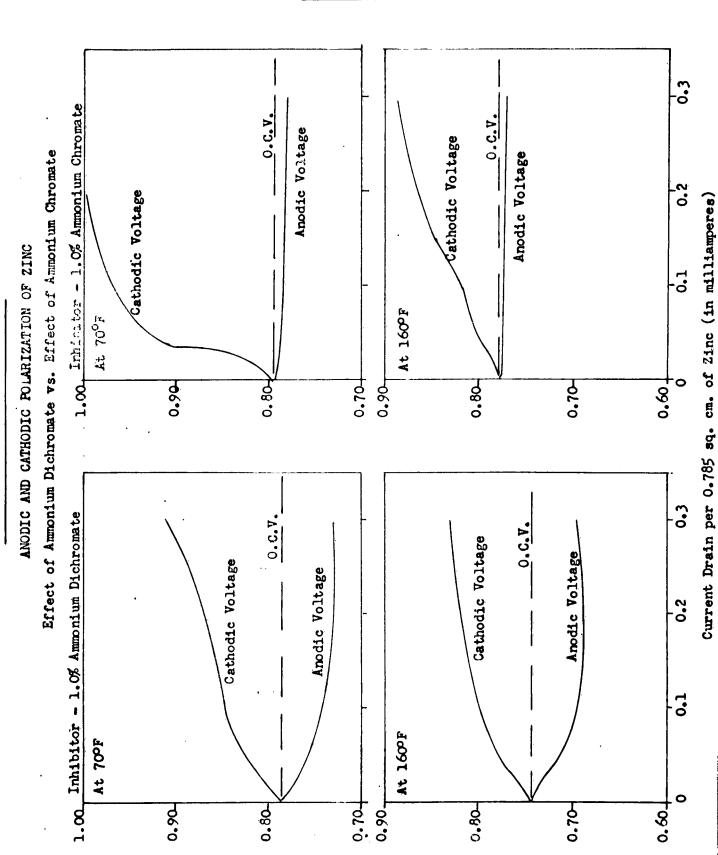
		CATHODIC	AND AN	ODIC POL	LARIZATI	ON		
Inhibitor	1.0%	(NH4)2Cr	207		1.0% (NH4)2Cr(04	
	Tested	€ 70°F	Tested (⊌ 160 ⁰ F	Tested (و 70 ° F	Tested	@ 160 ° F
	OCV -	0.795 v.	OCV -	0.753 v.	OCV - O	.798 v.	OCV - O	.779 v.
Polarization Current	Anode Voltage			Cathode Voltage		Cathode Voltage	Anode Voltage	Cathode Voltage
0.01 ma.	0.790	0.801	0.741	0.763	0.792	0.807	0.776	0.784
0.029 ma.	0.778	0.810	0.728	0.768	0.791	0.821	0.767	0.790
0.035 ma.	0.773	0.819	0.726	0.776	0.790	0.842	0.764	0.799
0.046 ma.	0.768	0.825	0.718	0.780	0.791	0.850	0.751	0.803
0.075 ma.	0.763	0.836	0.712	0.790	0.788	0.887	0.744	0.822
0.100 ma.	0.753	0.843	0.701	0.796	0.787	0.915	0.725	0.830
0.150 ma.	0.745	0.860	0.692	0.811	0.785	0.945	0.713	0.846
0.300 ma.	0.721	0.884	0.671	0.826	0.782	0.977	0.671	0.862

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Voltage of Anodically Polarized Zinc

FIGURE 6B (Cont.)



Voltage of Anodically and Cathodically Polarized Zinc

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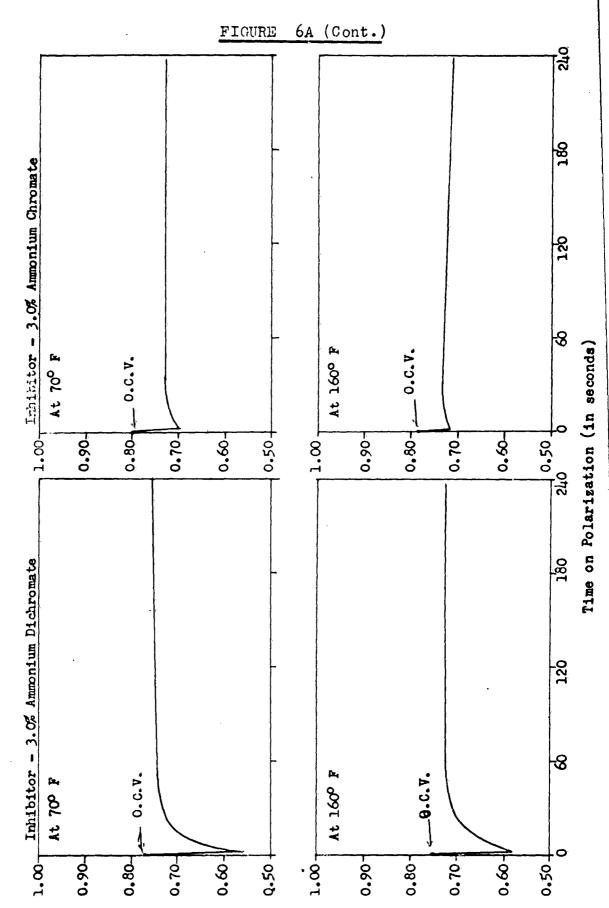
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FIGURE 6 (Cont.)

	ANODI	C POLARIZATIO	N ·					
Anodic Polarizati	lon Current De	nsity	4.33 ma./sq.	cm.				
Inhibitor	3.0% (NH4)2	Cr207	3.0% (NH4)2Cr	04				
	After 24 hr at 70°F	After 24 hrs at 160°F	After 24 hrs at 70°F	After 24 hrs at 1600F				
O.C.V. before polarization	0.772 v.	0.754 v.	0.795 v.	0.789 v.				
Time polarized		Anodic Voltage						
1 second	0.558 v.	0.578 v.	0.698 v.	0.718 v.				
15 "	0.713 v.	0.678 v.	0.724 v.	0.732 v.				
30 "	0.731 v.	0.711 v.	0.726 v.	0.735 v.				
	0.742 v.	0.725 v.	0.730 v.	0.734 v.				
90 h	0.747 v.	0.728 v.	0.731 v.	0.731 v.				
120 "	0.750 v.	0.729 v.	0.730 v.	0.728 v.				
180 "	0.754 v.	0.729 v.	0.729 v.	0.721 v.				
240 "	0.756 v.	0.729 v.	0.728 v.	0.716 v.				

	CATHODIC AND ANODIC POLARIZATION							
Inhibitor	Inhibitor 3.0. (NH4)2Cr2O7					H4)2Cr0	<u> </u>	
_	Tested	rested @ 70°F Tested @ 160°F		Tested	€ 70°F	Tested	@ 160°F	
).788 v.	OCV - (.756 v.	0CA - ().796 v.	OCV - ().783 v.
Polarization Current	Anode Voltage	Cathode Voltage	Anode Voltage	Cathode Voltage		Cathode Voltage		Cathode Voltage
0.01 ma	0.780	0.794	0.756	0.760	0.790	0.812	0.783	0.788
0.029 ma.	0.769	0.806	0.752	0.767	0.788	0.829	0.782	0.796
0.035 ma.	0.760	0.812	0.750	0.775	0.786	0.864	0.781	0.802
0.046 ma.	0.754	0.825	0.740	0.779	0.787	0.927	0.782	0.807
0.075 ma.	0.742	0.835	0.728	0.796	0.785	0.955	0.779	0.825
0.100 ma.	0.735	0.856	0.707	0.804	0.783	0.974	0.780	0.840
0.150 ma.	0.725	0.876	0.697	0.821	0.782	0,993	0.777	0.892
0.300 ma.	0.708	0.932	0.691	0.840	0.778	1.029	0.775	0.934

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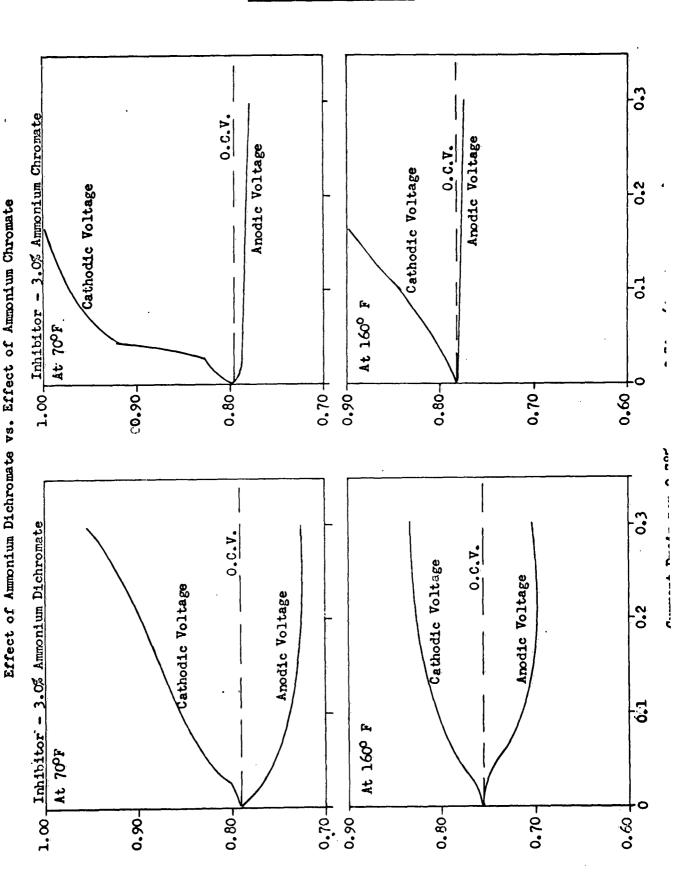


ANODIC POLARIZATION OF ZINC

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FIGURE 6B (Cont.)



ANODIC AND CATHODIC POLARIZATION OF ZINC

Voltage of Anodically and Cathodically Polarized zinc

FIGURE _7

	ANODI	C POLARIZATIO	ON				
Anodic Polarizat	ion Current De	n sity	4.33 ma./sq.c	·m•			
Inhibitor	0.25% Hg in	Zn	0.25% Hg in Zn	• 0.2% AmCr			
		After 24 hrs at 130°F	After 24 hrs at 70° F	After 24 hrs at 130°F			
O.C.V. before polarization	0.807 v.	0.801 v.	0.797 v.	0.799 v.			
Time polarized	Anodic Voltage						
l second.	0.778 v.	0.797 v.	0.658 v.	0.658 v.			
15 "	0.778 v.	0.779 v.	0.692 v.	0.624 v.			
30 "	0.777 v.	0.777 v.	0.678 v.	0.621 v.			
60 "	0.774 v.	0.778 v.	0.705 v.	0.622 v.			
90 "	0.773 v.	0.778 v.	0.709 v.	0.625 v.			
120 "	0.774 v.	0.778 v.	0.711 v.	0.628 v.			
180 "	0.776 v.	0.777 v.	0.719 v.	0.634 v.			
240 "	0.776 v.	0.777 v.	0.721 v.	0.635 v.			

	CATHODIC AND ANODIC POLARIZATION							
Inhibitor	0.25%	Hg in Z	n		0.25% на	g in Zn	+ 0.2%	AmCr
	Tested	@ 70°F	Tested	@ 130°r	Tested	@ 70°F	Tested	@ 130F
	OCV - C	.808 v.	OCV - C	.804_v.	OCV - C	0.800 v.	ocv - c	0.797 v .
Polarization Current	Anode Voltage	Cathode Voltage		Cathode Voltage		Cathode Voltage		Cathode Voltage
0.01 ma.	0.808	0.809	0.804	0.805	0.794	0.812	0.796	0.800
0.029 ma.	0.808	0.809	0.804	0.805	0.786	0.830	0.792	0.804
0.035 ma.	0.807	0.810	0.804	0.805	0.784	0.840	0.791	0.808
0.046 ma.	0.807	0.810	0.804	0.805	0.783	0.846	0.787	0.812
0.075 ma.	0.807	0.811	0.804	0.805	0.777	0.853	0.783	0.824
0.100 ma.	0.807	0.812	0.804	0.805	0.773	0.858	0.774	0.833
0.150 ma,	0.806	0.814	0.803	0.806	0.765	0.865	0.767	0.853
0.300 ma.	0.804	0.820	0.803	0.806	0.751	0.878	0.753	0.865

FIGURE 7 (Cont.)

	ANODIC	POLARIZATION						
Anodic Polarizat	ion Current De	nsity	4.33 ma./sq.c	om.				
Inhibitor	0.1% Antaro	n R 155	0.3% Antarox A	403				
•	After 24 hrs at 70° F	After 24 hrs at 130° F	After 24 hrs at 70° F	After 24 hrs at 130° F				
O.C.V. before polarization	0.754 v.	0.764 v.	0.780 v.	0.773 ₩.				
Time polarized		Anodic Voltage						
1 second	0.628 v.	0.738 v.	0.683 v.	0.716 v.				
15 "	0.646 v.	0.696 v.	0.722 v.	0.713 v.				
30 "	0.662 v.	0.700 v.	0.729 v.	0.714 v.				
60 "	0.681 v.	0.707 v.	0.735 v.	0.715 v.				
90 "	0.688 v.	0.711 v.	0.738 v.	0.718 v.				
120 ".	0.694 v.	0.714 v.	0.741 v.	0.721 v.				
180 "	0.701 v.	0.720 v.	0.742 v.	0.724 v.				
240 "	0.705 v.	0.724 v.	0.745 v.	0.724 v.				

	(CATHODIC	AND ANG	DDIC POL	ARIZATI	ON		
Inhibitor	0.1%	Antaron	R 155		0.3%	Antarox	A 403	
	Tested	@ 70 ° F	Tested	@ 130°F	Tested	@ 70°F	Tested	@ 130°)
	OCV - (0.771 v.	ocv - c	0.774 v.	ocv -	0.771 v.	ocv -	0.771 V
Polarization Current	Anode Voltage	Cathode Voltage		Cathode Voltage	Anode Voltage	Cathode Voltage	Anode Voltage	Cathode Voltage
0.01 ma.	0.754	0.807	0.767	0.777	0.765	0.809	0.757	0.780
0.029 ma.	0.741	0.838	0.761	0.780	0.759	0.835	0.755	0.783
0.035 ma.	0.734	0.843	0.759	0.795	0.757	0.842	0.754	0.784
0.046 ma.	0.731	0.852	0.755	0.817	0.754	0.849	0.754	0.786
0.075 ma.	0.724	0.865	0.749	0.830	0.748	0.863	0.751	0.791
0.100 ma.	0.717	0.879	0.741	0.833	0.743	0.874	0.748	0.796
0.150 ma,	0.716	0.894	0.732	0.837	0.737	0.890	0.745	0.811
0.300 ma.	0.704	0.876	0.721	0.845	0.722	0.892	0.737	0.825

FIGURE 7 (Cont.)

	ANOD	IC POLARIZATI	ON				
Anodic Polarizat	ion Current De	nsity	4.33 ma./sq.	cm.			
Inhibitor	0.2% AmCr + (0.1% Ant.R155	0.2% Hg in Zn	+ 0.1% Ant.R1			
	After 24 hrs at 700 F	After 24 hrs at 130° F	After 24 hrs at 70° F	After 24 hrs at 130° F			
0.C.V. before polarization	0.774 v.	0.744 v.	0.797 v.	0.798 v.			
Time polarized		Anode Voltage					
1 second ·	0.578 v.	0.453 v.	0.648 v.	0.718 v.			
15 "	0.631 v.	0.468 v.	0.674 v.	0.711 v.			
3 0 "	0.633 v.	0.468 v.	0.683 v.	0.713 v.			
60 "	0.638 v.	0.458 v.	0.690 v.	0.714 v.			
90 "	0.645 v.	0.461 v.	0.693 v.	0.714 v.			
120 "	0.650 v.	0.465 v.	0.696 v.	0.715 v.			
180 "	0.658 v.	0.467 v.	0.701 v.	0.717 v.			
240 ⁿ	0,662 v.	0.468 v.	0.703 v.	0.717 v.			

	· CA	THODIC	AND ANOI	DIC POLA	RIZATIO	N		
Inhibitor	0.2% Am	r + 0.1	% Ant. F	₹155	0.2% Hg	in Zn +	0.1% Ar	nt.R155
	Tested @	70°F	Tested	@ 130°F	Tested	@ 70°F	Tested @	130°F
	OCV - C	.780 v.	OCV - C	.775 v.	OCV - (0.78 6 v.	OCV - (0.779 v
Polarization Current	Anode Voltage	Cathode Voltage		Cathode Voltage	l	Cathode Voltage		Cathod Voltag
0.01 ma.	0.766	0.813	0.770	0.814	0.743	0.874	0.773	0.843
0.029 ma.	0.753	0.845	0.756	0.840	0.734	0.897	0.764	0.859
0.035 ma.	0.750	0.851	0.750	0.852	0.732	0.898	0.762	0.859
0.046 ma.	0.747	0.861	0.746	0.871	0.732	0.900	0.756	0.865
0.075 ma.	0.739	0.881	0.730	0.894	0.727	0.887	0.748	0.883
0.100 ma.	0.732	0.895	0.732	0.898	0.729	0.891	0.739	0.891
0.150 ma.	0.718	0.908	0.708	0.897	0.725	0.892	0.729	0.899
0.300 ma.	0.696	0.948	0.700	0.915	0.721	0.902	0.707	0.892

FIGURE 7_(Cont.)

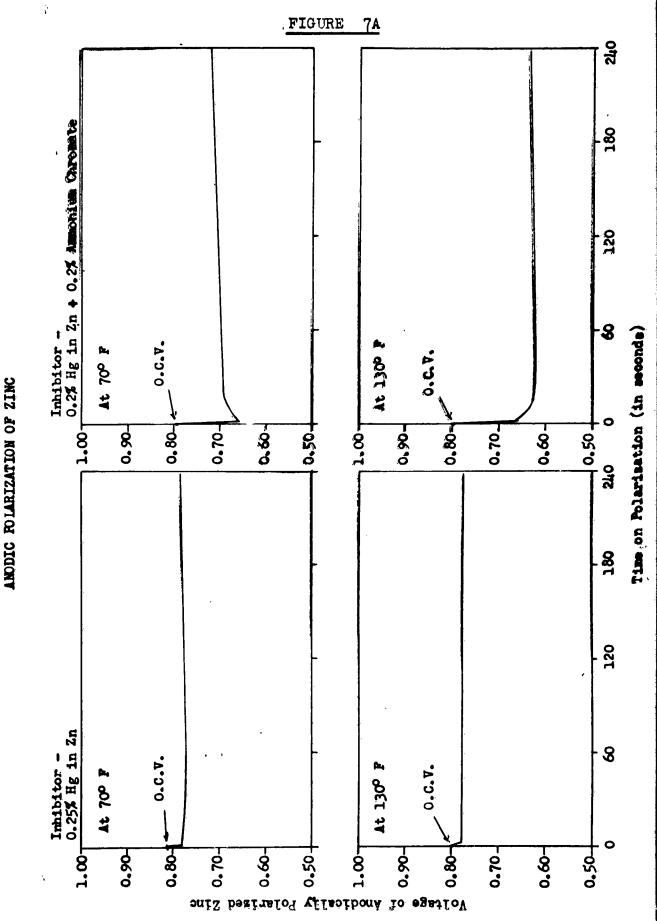
	ANODIC	POLARIZATION		
Anodic Polarizat	tion Current De	nsity	4.33 ma./sq.c	n.
Inhibitor	0.2% AmCr +	0.3% Ant.A403	0.2%Hg 1n + 0	.3% Ant. A403
	After 24 hrs	After 24 hrs at 130° F	After 24 hrs. at 70° F	After 24 hrs at 130° F
0.C.V. before polarization	0.790 v.	0.769 v.	0.807 v.	0.801 v.
Time polarized		Anode	Voltage	
1 second	0.613 v.	0.683 v.	0.753 v.	0.796 v.
15 "	0.629 v.	0.698 v.	0.753 v.	0.787 v.
30 "	0.626 v.	0.705 v.	0.752 v.	0.786 v.
60 "	0.616 v.	0.713 v.	0.754 v.	0.786 v.
90 "	0.605 v.	0.717 v.	0.756 v.	0.787 v.
120 !"	0.596 v.	0.719 v.	0.757 v.	0.786 v.
180 "	0.585 v.	0.721 v.	0.758 v.	0.787 v.
240 "	0.578 v.	0.723 v.	0.760 v.	0.787 v.

	C	ATHODIC	AND ANO	DIC POLA	ARIZATIO	N		
Inhibitor	0.2° .m	Cr + 0.3	3% Antar	ox A403	0.2% Hg	in Zn	0.3% A	nt.A403
	Tested	@ 70 ° F	Tested	© 130 0 F	Tested	© 70 ° F	Tested	& 130 ^o f
	OCV - O	OCV - 0.796 v. OCV - 0.766 v. OC		OCV - O	.808 v.	OCV - O	.802 v.	
Polarization Current	[Cathode Voltage		Cathode Voltage	Anode Voltage	Cathode Voltage		Cathode Voltage
0.01 ma.	0.787	0.798	0.748	0.722	0.807	0.808	0.802	0.802
0.029 ma.	0.774	0.814	0.747	0.778	0.807	0.809	0.802	0.802
0.035 ma.	0.771	0.821	0.747	0.783	0.807	0.810	0.802	0.803
0.046 ma.	0,766	0.825	0.746	0.793	0.807	0.810	0.802	0.803
0.075 ma.	0.756	0.839	0.745	0.805	0.806	0.811	0.802	0.803
0.100 ma.	0.752	0.848	0.742	0.819	0.806	0.813	0.802	0.804
0.150 ma.	0.743	0.873	0.740	0.835	0.805	0.816	0.802	0.804
0.300 ma.	0.735	0.887	0.739	0.865	0.803	0.825	0.802	0.806

FIGURE 7_(Cont.)

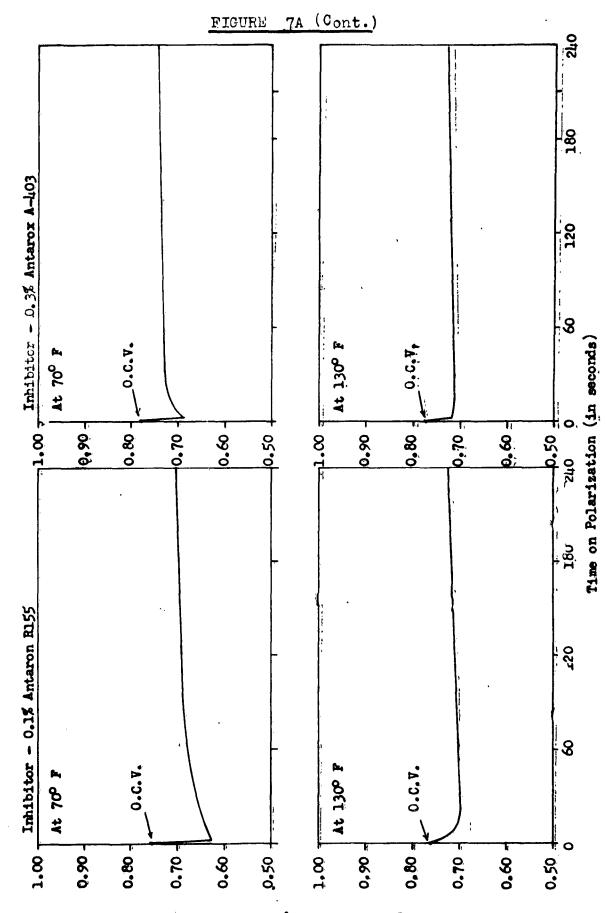
	ANODIC	POLARIZATIO	N					
Anodic Polarizat	tion Current De	nsity	4.33 ma./sq.	cm.				
Inhibitor	0.2% AmCr +	0.3% Neut.	0.2% Hg in Zn	+ 0.3% Neut.				
	After 24 hrs	After 24 hr at 130° F	After 24 hrs. at 70° F	After 24 hrs. at 130° F				
0.C.V. before polarization	0.768 v.	0.766 v.	0.806 v.	0.798 ▼.				
Time polarized		Anodic Voltage						
1 second	0.663 v.	0.6 5 8 v.	0.718 v.	0.761 v.				
15 "	0.703 v.	0.656 v.	0.7 20 v.	0.744 v.				
30 "	0.707 v.	0.655 v.	0.723 v.	0.743 v.				
60 "	0.712 v.	0.653 v.	0.728 v.	0.742 v.				
90 "	0.714 v.	0.656 v.	0.732 v.	0.743 v.				
120 ".	0.715 v.	0.656 v.	0.735 v.	0.743 v.				
180 "	0.715 v.	0.657 v.	0.739 v.	0.743 v.				
240 "	0.716 v.	0.658 v.	0.741 v.	0.743 v.				

	Cathodic and Anodic Polarization								
Inhibitor	0.2% Am	Cr + 0.	3% Neutr	onyx	0.2% He	in Zn	+ 0.3% Neutron		
	Tested	@ 70°F	Tested @ 130°F		Tested	© 70 0 F	Tested	@ 130°F	
	OCV - O	.778 v.	OCV - C	.756 v.	OCV - C	.807 v.	OCV - C	.802 v.	
Polarization Current	Anode Voltage	Cathode Voltage		Cathode Voltage	1	Cathode Voltage		Cathode Voltage	
0.01 ma.	0.776	0.798	0.742	0.787	0.807	0.808	0.802	0.802	
0.029 ma.	0.774	0.820	0.736	0.823	0.805	0.811	0.802	0.803	
0.035 ma.	0.771	0.830	0.735	0.831	0.804	0.812	0.802	0.803	
0.046 ma.	0.766	0.837	0.733	0.839	0.803	0.813	0.802	0.803	
0.075 ma.	0.758	0.852	0.727	0.852	0.801	0.817	0.802	0.804	
0.100 ma.	0.751	0.862	0.730	0.865	0.799	0.820	0.801	0.804	
0.150 ma.	0.742	0.883	0.724	0.882	0.796	0.827	0.801	0.804	
0.300 ma.	0.721	0.903	0.716	0.887	0.788	0.850	0.799	0.806	



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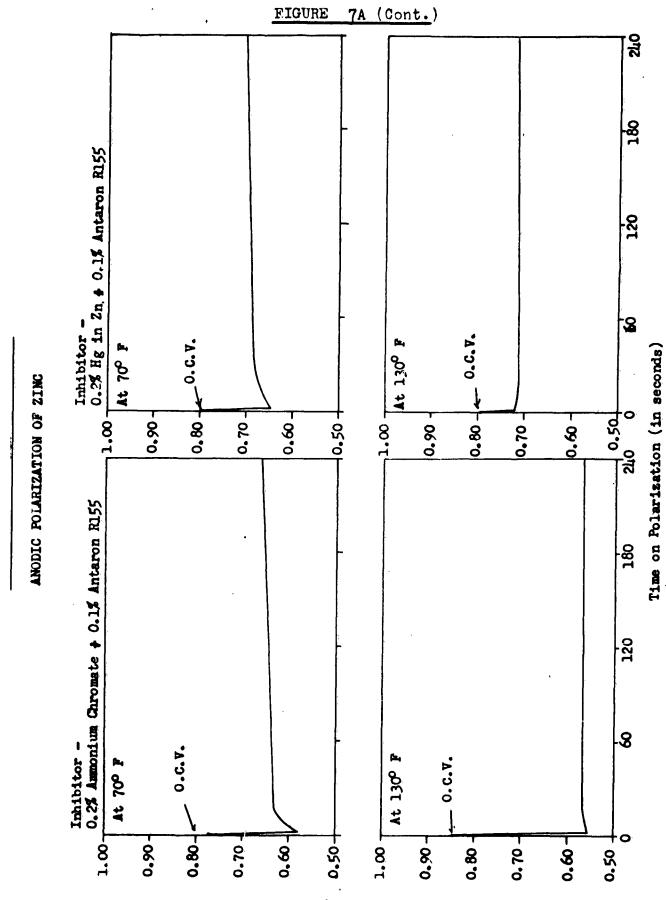


ANDIC POLARIZATION OF ZINC

Voltage of Amodically Polarised Zinc

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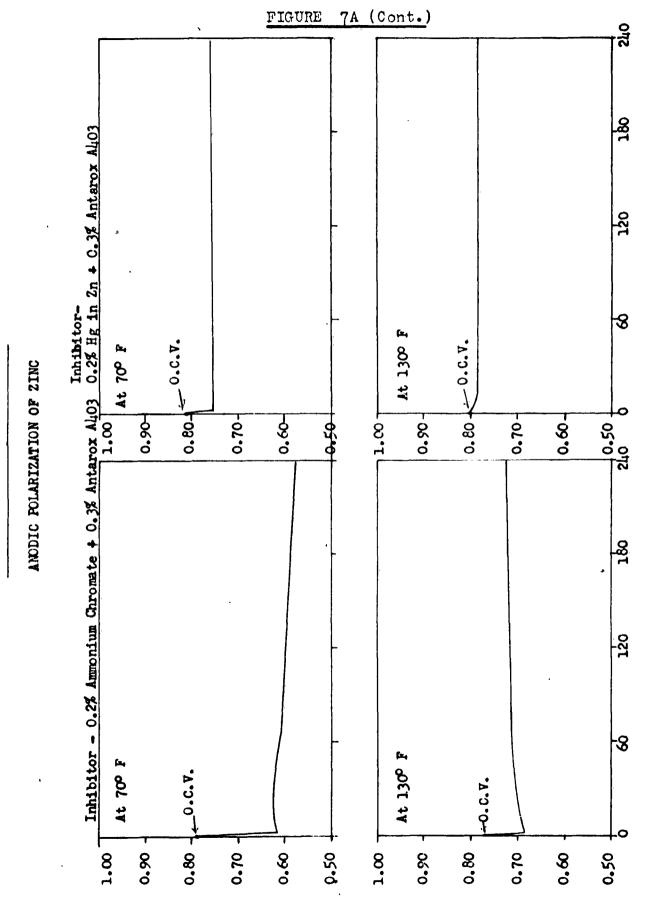
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Voltage of Anodically Polarized Zinc

Time on Polarization (in seconds)

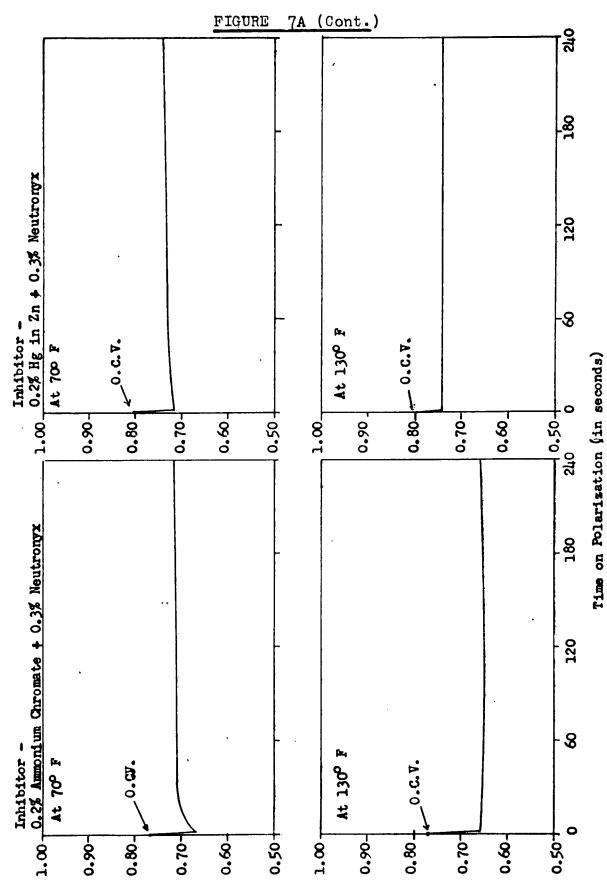
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Voltage of Anodically Polarized Zinc

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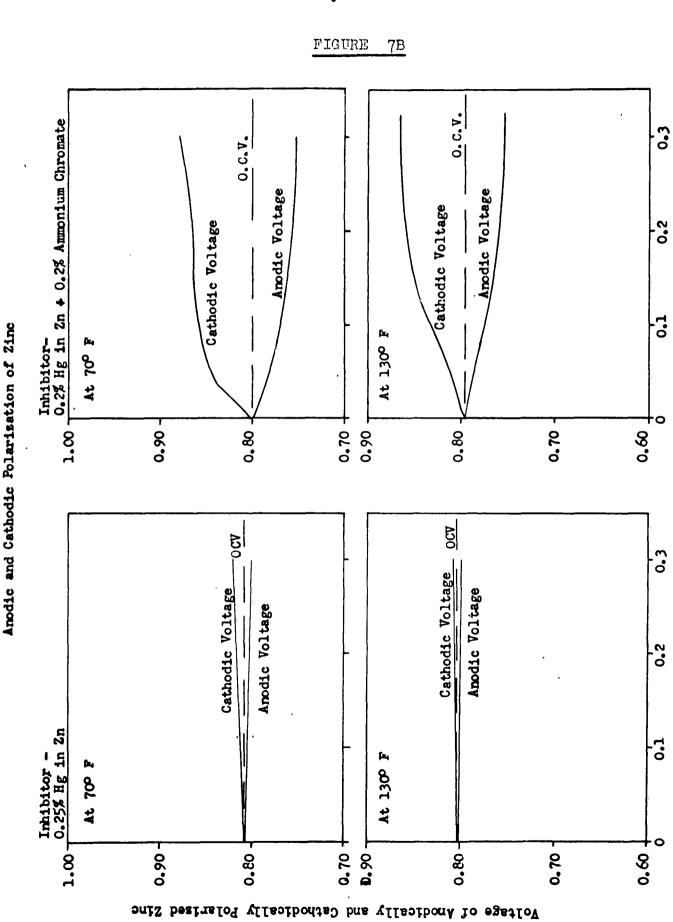


ANDLIC POLARIZATION OF ZINC

Voltage of Amodically Polarized Zinc

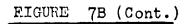
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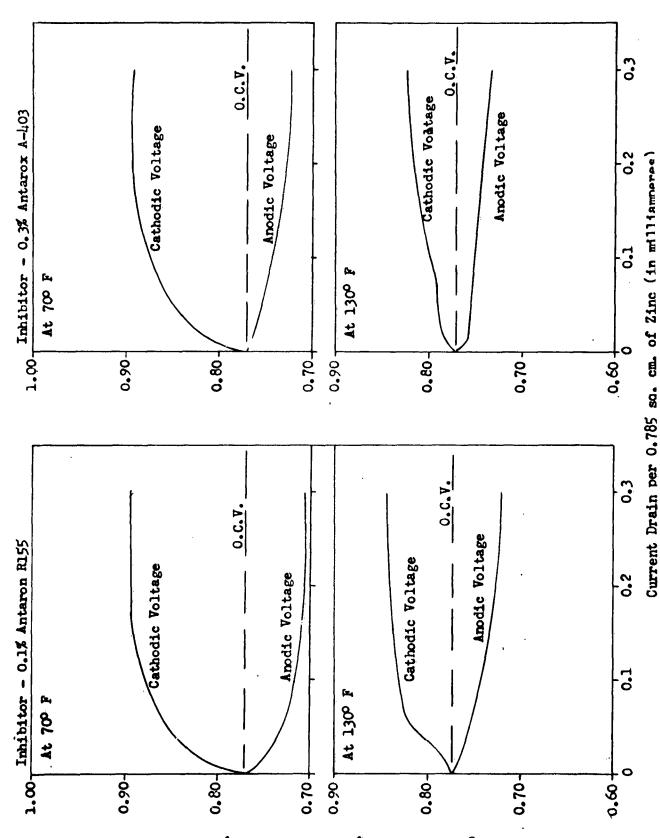
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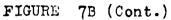
ANDDIC AND CATHODIC POLARIZATION OF ZINC

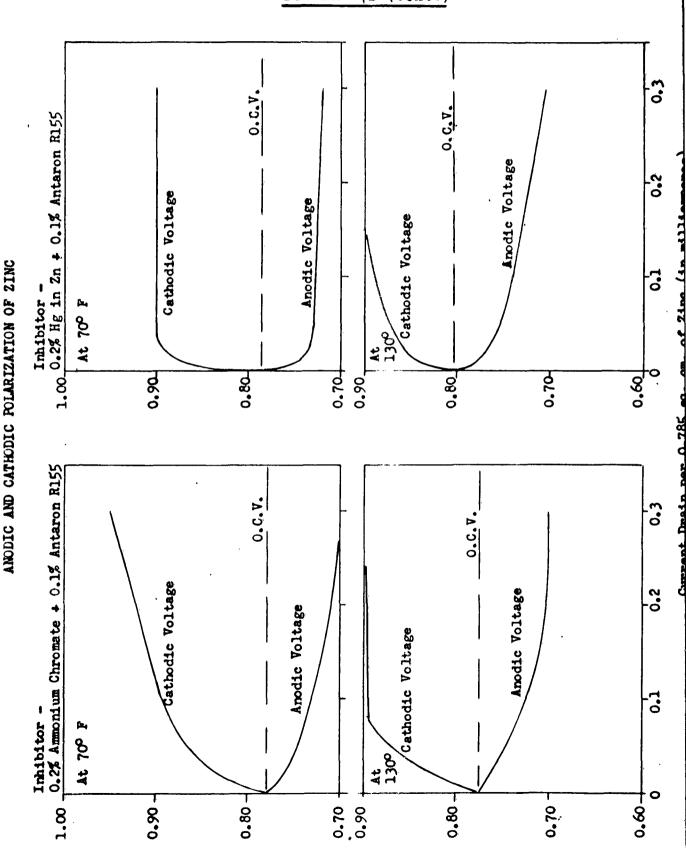
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 $\dot{V}\text{oltage}$ of Anodically and Cathodically Polarized Zinc

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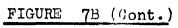
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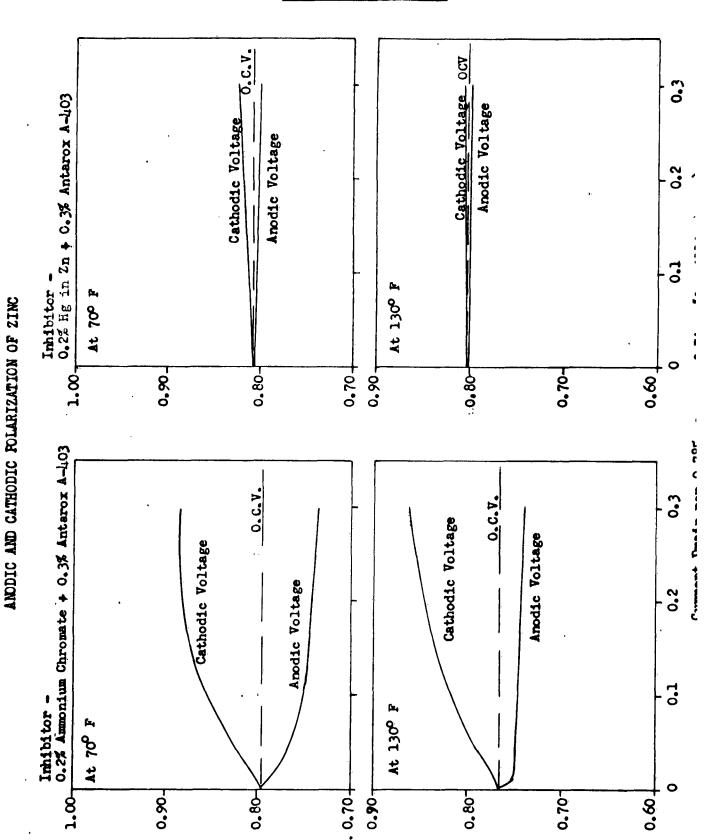




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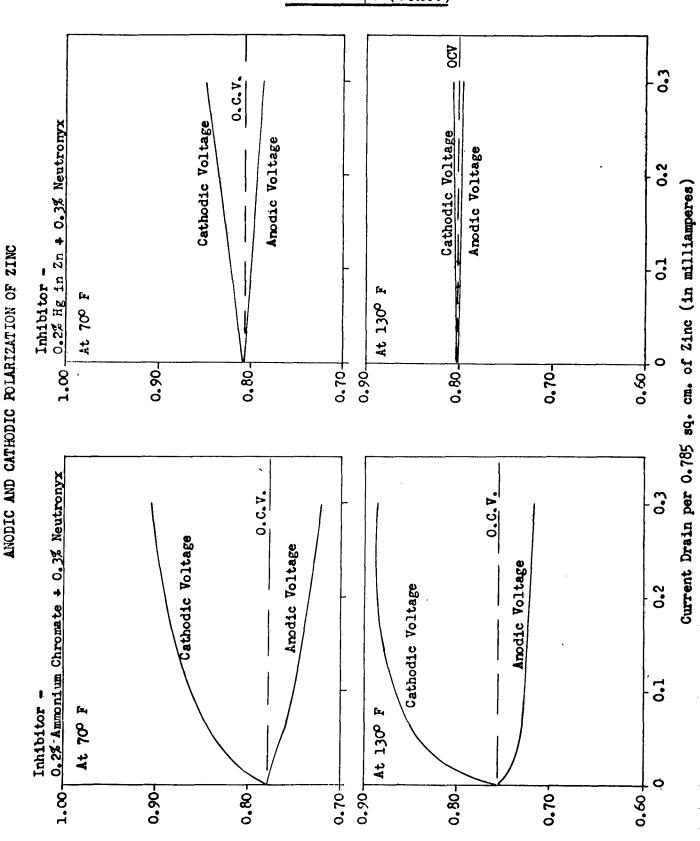




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FIGURE 7B (Cont.)



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Voltage of Anodically and Cathodically Polarized Zinc

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PIGURE 8	3
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Visual Corrosion Studies (Linc partially immersed)

Storage Conditions.

Strip of zinc stored in electrolyte containing inhibitor. Inhibitor percentage by weight of electrolyte. Sample stored at 1130 F for one year.

Sample		Percent	weight of	zinc	Percent
No.	Inhibitor	of Inhibitor	Initially	After 12 mos at 113 ⁰ F	
1		0.05%	1.20 gm.	0.93 gm.	22.5%
2	Antarox A-200	0.20	1.21	0.95	21.5
3		0.50	1.33	1.05	21.1
4		0.05	1.27	0.98	22.8
5	Antarox D-100	0.20	1.45	1.06	26 .7
6		0.50	1.05	0.81	22.8
7		0.05	1.16	0.87	25.0
8	Antarox A-403	0.20	1.24	0.91	26.6
9		0.50	1.20	0.91	24.1
10		0.05	1.35	1.06	21.5
11	Antarox A-400	0.20	1.10	0.82	25.4
12		0.50	1.20	0.99	17.5
13		0.05	1.29	1.00	22.5
14	Quaternar "C"	0.20	1.01	0.77	24.7
15		0.50	1.15	0.96	16.5
16		0.05	1.28	1.07	16.4
17	Amine "C"	0.20	1.11	0.93	16.2
18		0.50	1.07		
19		0.05	1.30	1.07	17.7
20	Antaron R-155	0.20	1.16	0.99	14.6
21		0.50	1.19	1.00	16.0
2 2		. 0.05	1.33	1.06	20.3
23	Detergent D-60FG	0.20	1.16	0.90	22.4
24		0.50	1.03	0.92	10.7
25	"	0.05	1.09	0.88	19.3
26	Sequestrene NA-2	0.20	1.08	0.67	37.0
27		0.50	1.13	0.79	30.1

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FIGURE 8 (cont.)

Sample		rercent	Veight of	zinc	Percent
No.	Inhibitor	of inhibitor	Initially	After 12 mos 113°F	of weight lost
28		0.05	1.19 gm.	0.98 gm.	17.6%
29	Sequestrene NA-4	0.20	1.30	1.11	14.6
30		0.50	1.10		
31		0.05	1.21	1.00	17.4
32		0.20	1.16	1.05	9.5
33	Na2Cr2O7	0.50	1.43	1.34	6.3
34		1.00	1.32	1.27	3.8
. 35		2.00	1.08	1.05	2.8
36		0.50	1.24	0.98	21.0
37	Denatured Gluten	1.00	1.37		
-3 8		2.00	1.35		
39	Special Wheat	0.50	1.18	0.90	23.8
40	Protein R2E-570	1.00	1.32	1.21	8.4
41		2.00	1.05		
42		0.50	1.19		
43	Protein #3323	1.00	1.33		
44		2.00	1.30		
45	Devitalized	0.50	1.14		
46	Wheat Gluten	1.00	1.15	0.920	20.0
47		2.00	1.19		
48		0.50	1.28		
49	Triticote #87	1.00	1.21		
50		2.00	1.11	0.81	27.0
51	Special Wheat	0.50	1.24	1.00	19.3
52	Protein R2E-1301	1.00	1.05		
53		2.00	1.41	1.09	22.7
54	Mercury in zinc	0.05	1.22	0.99	18.8
55	hercury in zinc	0.10	1.27	1.00	21.6
56	Mercury in zinc	0.20	1.16	0.89	23.2
57	Mercury in zinc	0.30	1.14	0.76	33.3
58	Mercury in zinc	0.40	1.26	0.67	47.0

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FIGURE 9	}

Visual Corrosion Studies (Linc totally immersed)

Storage
Conditions

Strip of zinc stored in electrolyte containing inhibitor. Inhibitor percentage by weight of electrolyte.

Sample stored at 115°F for one year.

Jample	The state of the s	rercent	eizut	of zinc	Fercent
NO.	Inhibitor	of Inhibitor	Initially	mos 113°F	of weight lost
l		0.05,0	0.84 gm.	0.71 gm.	15.5%
2	Antarox A-200	0.20	0.60	0.46	25.3
3		0.50	0.47	0.39	17.0
4		0.05	0.30	0.66	17.5
5	Antarox D-100	0.20	0.71	0.54	24.0
6		0.50	0.90	0.68	15.0
7	•	0.05	0.75	0.61	18.7
8	Antarox A-403	0.20	0.59	0.48	18.7
9		0.50	U.63	0.57	9.5
10		0.05	U.80	0.68	15.0
11	Antarox A-400	0.20	0.65	0.51	21.6
12		0.50	0.64	0.52	18.8
13	 	0.05	0.89	0.71	20.0
14	uaternary "C"	೦ ಜ೦	0.71	0.58	18.3
15		0.50	U. 37	0.72	17.2
16		0.05	0.78	0.58	25.6
17	Amine "C"	0.20	0.78	0.67	14.1
18		0.50	0.77	0.69	10.4
19		0.05	0.82	0.74	9.8
20	Antaron R-155	0.20	0.70	0.61	12.8
21		0.50	0.71	0.63	11.3
22		0.05	0.78	0.67	14.1
23	Detergent D-60FG	0.20	0.74	0.68	8.1
24]	0.50	0.71	0.53	25.4
. 25		0.05	0.80	0.62	22.5
20	Sequestrene MA-2	0.20	0.72	0.60	16.7
27	-	0.50	.0.80	0.68	15.0

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FIGURE 9 (cont.)

Sample No/	Inhibitor	rercent of Inhibitor	weight of	zinc	Percent
			Initially	After 12 mos113°F	of weight
28	Sequestrene NA-4	0.05%	0.86 gm.	0.61 gm.	29.0%
29		0.20	0.81	0.61	24.8
3 0		0.50	0.90	0.62	31.1
31	NagCrgO7	0.05	0.87	0.81	6.9
32		0.20	0.79	0.74	6.3
33		0.50	0.73	0.69	5.5
34		1.00	0.71	0.68	4.2
35		ఓ.00	0.65	0.62	4.6
36	Denatured Gluten	0.5)	0.75	0.66	12.0
37		1.70	0.77	0.65	15.6
38		2.00	0.81	0.69	14.8
39	Special Wheat	0.50	0.74	0.70	5.4
40	Protein R2E-570	1.00	0.70	0.65	7.2
41		2.00	0.70	0.60	14.3
42	Protein #3323	0.50	0.65	0.57	18.5
43		1.00	0.73	0.64	12.3
44		2.00	0.64	0.52	18.8
45	Devitalized Wheat	0.50	0.76	0.70	7.9
46	Gluten	1.00	0.82	0.63	8.53
47		2.00	0.72	0.59	18.1
48	Triticote #87	0.50	0.68	0.59	13.2
49		1.00	0.77	0.64	16.9
50		2.00	0.79	0.62	21.5
51	Special Wheat	. 0.50	0.71	0.63	11.3
52	Frotein R2E-1301	1.00	0.75	0.63	16.0
53		2.00	0.76	0.69	9.2
54	Lercury in zinc	0.05	0.65	0.52	20.0
55	Mercury in zinc	0.10	0.81	0.66	18.5
56	Lercury in zinc	0.20	0.81	0.57	29.6
57	Mercury in zinc	0.30	0.66	0.44	33.4
58	Lercury in zinc	0.40	0.54	0.13	76.0

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PART VI

CONSTRUCTION AND
CAPACITY RESULTS
OF STORED CELLS

Part VI - Capacity Results of Cells Stored at the Temperatures Specified by the Contract

Exploratory experiments have been done in an effort to determine what structural changes must be made in conventional cells in order that they may meet the specifications required by this contract, namely, storage twelve months at 113°F, 95% R.H., three months at 130°F, 50% R.H. and two weeks at 160°F, 50% R.H. After this storage period, it is desired that the cells have capacities at least one half of the initial capacities of corresponding cells.

Some satisfactory data have been obtained but the problem of subseal leakage was a serious one. The subseal used was a high melting form of Amprol wax (Atlantic Refining Company). This wax does not normally flow at 160°F but under the pressure generated in cells stored at 160°F it is forced out around the top seal.

In order to overcome this problem several different types of waxes were tested, but none was found which could be handled satisfactorily and still not result in subseal leakage. Another method was found however that decreased the subseal and paste leakage considerably and, in many cases, stopped it completely. This method involved the use of a polyethylene gasket incorporated into the top seal design. This gasket was crimped between the zinc can and the plastic top seal making a tight seal at this point and at the same time it gave a tight seal between the top seal and the carbon rod. This gasket was used with and without a wax subseal and the results were compared to those using a conventional amprol wax subseal. These results are shown in Figure #1. A heat resistant polystyrene cap has been used as the final top seal and has proven to be very satisfactory. This cap is held in place by crimping the top of the zinc can.

Several lots of fabricated cells contained 100% African ore mixes in which conventional inhibitors were tested. Both conventional starch formulations (corn starch, potato starch and flour) and modified starch formulations were used. Other lots were made in which 100% Cryptomelane and 30% Cryptomelane - 70% African ore mixes were employed. Several types (morphological) of Cryptomelane were tried in the latter tests. A conventional separator of corn starch, potato starch, flour and electrolyte and a mercury inhibitor were used in those cells containing Cryptomelane ores.

The best results were obtained with cells using sodium dichromate as the inhibitor. In the cases where the dichromate was used as the inhibitor a reservoir of chromate ions was added to the mix in the form of chromic oxide or barium chromate. Since the ore has a tendency to absorb chromate ions, this reservoir was added to satisfy the absorption rather than taking the ions from the inhibitor in the paste.

In many cases it has been noticed that the use of chromate as an inhibitor seriously decreases delayed room temperature capacity due to the passivation of the zinc by the chrome film. The reason that excellent elevated temperature

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capacity is obtained may be that the passivating tendency of the chrome film is decreased. This has been shown to be true from polarization studies.

The use of chromic oxide or barium chromate as the ion reservoir does, however, make considerable difference. Cells using chromic oxide gave very poor results while those using barium chromate gave very good results. This has been also found to be true with cells stored at 160°F.

The capacity results obtained from cells at 130°F. show, as did the results obtained from cells stored at 160°F. and 113°F., that African ore appears to give more satisfactory and consistent results than do blends of African and an activated ore probably due to the fact that the African ore undergoes less change in crystal structure during the storage period than do the activated ores.

The evaluation of Anodite and Cryptolite as the depolarizer for high temperature shows that best results are obtained when they are blended with African ore (30% Cryptolite or Anodite - 70% African ore).

Capacity results of fabricated cells after storage are condensed and tabulated under storage temperature. Figures 2, 3 and 4 are for those cells which were stored twelve months at 113°F, 95% R.H., while Figures 5 and 6 are for cells stored three months at 130°F, 50% R.H., and Figures 7 and 8 are for cells stored two weeks at 160°F, 50% R.H.

Conclusions

- 1. Capacity results of cells stored at temperatures specified by the contract shows that:
 - a. Cells can be made which operate satisfactorily after storage for one year at 113°F. 95% R.H.
 - b. Cells can be made which operate satisfactorily after storage for each year at 130°F. 50% R.H.
 - c. Cells can be made which operate satisfactorily after storage for two weeks at 160°F. 50% R.H.
- 2. African ore appears to be the most satisfactory manganese dioxide for elevated temperature storage especially when cells are stored for an extended period.
- 3. Increased capacity and decreased leakage can be obtained from cells stored at 160°F. by the use of a polyethylene gasket, substituted for the wax sub-seal, and incorporating it as part of the heat resistant polystyrene top seal.
- 4. Using Cryptomelane in blends with African ore gave excellent results after storage at 113°F. 95% R.H. for a period of one year.
- 5. Best capacity results from cells stored at either 130°F. or 160°F. were obtained from those using sodium dichromate, a combination of sodium dichromate and Antaron R-155 or a combination of mercury and Antaron R-155 as the inhibitor.

TABLES AND RESULTS

PART VI

CONSTRUCTION AND CAPACITY RESULTS OF CELLS

STORED AT THE SPECIFIED TEMPERATURES OF

THE CONTRACT.

Comparative Capacity Results Of Cells Stored At 160°F

	conditions before	ore	2 weeks at 70°F,	50% R.H.		
Storage stored s 70°F)	conditions for at 160°F (tested	cells at	2 weeks at 70°F 2 weeks at 160°F	2 weeks at 70°F, 50% R.H. 2 weeks at 160°F, 50% R.H.		
			Type of Subseal	·		
Test	Testing Time	Amprol wax	Amprol wax with polyethylene gasket	Polyethylene gasket - no wax subseal		
	Initially	13,.7	13.7	13.7		
BA-30 (days)	After 2 wks. at 160°F	5.7	11.5	11.0		
BA-8	Initially	244.0	244.0	244.0		
(hrs.)	After 2 wks. at 160°F	143.0	180.4	168.8		
54.400	Initially	22.6	22.6	22.6		
BA-408 (hrs.)	After 2 wks. at 160°F	11.6	16.8	17.8		

CONSTRUCTION AND CAPACITY RESULTS OF CELLS

STORED AT

113° F.

FOR 1 YEAR AT 95% R.H.

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FIGURE	2	

LeClanche Type "D" Cells For Storage At 1130 F. (Construction)

	age Time Before Initial ing At 70° F.		2 weeks at 70°F - 50% R.H.			
Store At 1	age Time For Cells Stor 13°F (Tested at 70°F)	ed	l month at 70°F l year at 113°F	- 50% R.H - 95% R.H	•	
Lot No.	Mix Formulation	S	Separator	Subseal	Seal	
B2148	100% African Ore 7/1 - ore/black	0.3%	ch - flour paste HgCl ₂ as bitor	Amprol wax	Bakelite cap	
B2149	100% African ore 7/1 - ore/black	.0.3%	ch - flour paste HgCl ₂ as bitor	Amprol wax	Bakelite cap	
B2150	100% African ore 7/1 - ore/black 0.5% CrO3	0.3%	ch - flour paste Na2Cr2O7 as bitor	Amprol wax	Bakelite cap	
B2151	100% African ore 7/1 - ore/black 1.0% BaCr04	0.3%	ch - flour paste Na20r207 as bitor	amprol wax	Bakelite cap	
B2152	100% African ore 7/1 - ore/black 1.0% BaCr04	star	ially treated ch - 0.3% r ₂ 07 as inhibitor	amprol wax	Bakelite cap	
B2473	100% African ore 7/1 - ore/black 0.5% Cr03	star	cially treated cch - 0.3% Gr207 as inhibitor	amprol wax	Bakelite cap	
B2474	100% African ore 7/1 ore/black 0.5% CrO3	1	ch - flour paste were Gronak ited	amprol wax	Bakelite cap	
B2475	100% African ore 7/1 - ore/black 1.0% BaCrO4		ial starch paste were Cronak ited	amprol wax	Bakelite cap	

FIGURE 2 (cont.)

Lot No.	Mix Formulation	Separator	Subseal	Seal
B2476	100% African ore 7/1 - ore/black 1.0% Bacr04	Starch - flour paste Cans were Cronak treated	amprol wax	Bakelite cap
B2477	100% African ore 7/1 - ore/black	Starch - flour paste 0.5% Antarox A-403 as inhibitor	amprol wax	Bakelite cap
B2478	100% African ore 7/1 - ore/black	Starch - flour paste (0.3% HgCl2 as inhibitor	amprol wax	Bakelite cap
B2479	100% African ore 7/1 - ore/black 0.5% Cr03	Starch - flour paste 0.3% Na2Cr2O7 as inhibitor	amprol wax	Bakelite cap
B2480	100% African ore 7/1 - ore/black 1.0% BaCrO4	Starch - flour paste 0.3% Na2Cr2O7 as inhibitor	amprol wax	Bakelite cap
B2611	20% Light Hydrate 80% African ore 7/1 - ore/black	Special starch paste 0.3% HgCl ₂ as in- hibitor	amprol wax	Bakelite cap

FIGURE 2 A

LeClanche Type "D" Cells Stored at 113° F (Capacity)

	Test Results							
Lot		BA-30 (d	BA-30 (days)		urs)	BA-408/U (hours)		
No.	Cell No.	Initial	l year at	Initial	l year at	Initial	l year at	
B2148	1 2 3	13.7 13.5	4.6 5.0 5.0	193.0 212.0 200.0	96.0 104.0 88.0	19.0 19.0 19.0	12.0 10.7 12.6	
·	Ave.	13.6	4.9	202.0	96.0	19.0	11.8	
B2149	1 2 3	12.8 13.2 13.4	8.4 8.2 10.2	205.0 206.0 206.0	112.0 126.4 129.6	17.0 17.0 17.0	11.3 11.1 13.5	
	Ave.	13.1	8.9	206.0	122.6	17.0	11.8	
B2150	1 2 3	14.4 14.6 14.4	Cells no good	207.0 210.0 206.0	Cells no good	18.0 17.0 18.0	Cells no good	
	Ave.	14.5		208.0		17,7		
B2151	1 2 3	14.2 13.6 13.2	8.0 5.8	196.0 219.0 208.0	150.4 130.7 147.2	17.0 17.0 16.0	13.5 14.1 14.7	
	Ave.	13.7	,6.9	208.0	. 143.0	16.6	14.1	
B2152	1 2 3	13.6 13.6 14.0	1.5	195.0 195.0 192.0	Cells no good	16.0 17.0 17.0	14.0	
	Ave.	13.7	1.5	194.0		16.6	14.0	
B2473	1 2 3	14.4 14.0 12.8	10.0 10.2 10.8	194.0 195.0 194.0	133.1 146.3 137.4	18.0 18.0 17.0	14.8 16.0 16.6	
	Ave.	13.7	10.4	194.0	138.9	17.6	15.8	
B2474	1 2 3	13.6 14.0 13.0	Cells no good	195.0 208.0 200.0	Gells no good	18.0 17.0 16.0	Cells no good	
	AVe.	13.5		201.0		17. 0		
B2475	1 2 3	12.2 12.2 12.4	8.4 7.8 7.8	193.0 183.0 194.0	138.2 139.4 140.8	18.0 17.0 17.0	13.6 14.1 15.1	
	Ave.	12.3	8.0	196.0	139•5	17.3	14.3	

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FIGURE 2A (cont.)

	Test Results							
Lot		BA-30 (days)	BA-8 (hours)		BA-408/U (hours)		
No.	Cell No.	Initial	After 1 yr1130.	Initial	l year at 113°F	Initial	l year at 113°F	
B2476	1 2 3	13.8 13.0 13.0	6.2 6.0 7.2	18 4 200 200	126.4 125.1 130.0	17.0 17.0 17.0	13.6 15.2 15.6	
	Ave.	13.3	6.5	195	127.2	17.0	14.8	
B2477	1 2 3	13.0 13.0 13.8	4.6 7.4 7.6	208 203 192	119.4 128.0 112.0	15.0 15.0 15.0	11.1 13.3 11.8	
	Ave.	13.3	6,5	201	119.8	15.0	12.1	
B2478	1 2 3	12.6 12.6 13.4	9.2 8.6	192 203 183	75.2 126.4 108.0	15.0 16.0 15.0	14.2 10.3 17.2	
	Ave.	12.9	8.9	193	103.2	15.3	13.9	
B2479	1 2 3	12.6 15.0 13.0	Cells no good	192 189 183	Cells no .good	17.0 18.0 19.0	Cells no good	
	Ave.	13.5	~~~~	1 88		18.0		
B2480	2 3	19.0 19.0 19.2	8.8	200 208 192	124.0 120.7 124.0	16.0 16.0 16.0	14.7 14.9 12.0	
	Ave.	19.1	8.8	200	122.9	16.0	13.9	
B2611	2 3	14.4 12.8 13.4	Cells no good	230 246 232	Cells no good	27.0 28.0 30.0	Cells no good	
	Ave.	13.5		236		28.3		

$\mathbf{F}'\mathbf{I}$	GUR	U	3

Leclanche Type "D" Cells Stored At 113° F. (Construction)

	ge time before initial ag at 70° F.	2 weeks at 70°F - 50% R.II.			
	ge time for cells stored SOF (Tested at 7 0°F)	1 month at 70°F - 50% 1 year at 113°F - 95%	к.н. К.н.		
Lot No.	Mix Formulation	Separator	Subseal	Seal	
B 3178	100% African ore 7/1 - ore/black	Olin Starch Formulation #2 - 0.3% NagCr207 as inhibitor	Amprol wax	Bakelite cap	
B3179	100% African ore 7/1 - ore/black	Olin Starch Formulation #3 - 0.25% HgCl2 as inhibitor	li .	II .	
B3180	20% Light Hydrate 80% African ore 7/1 - ore/black	Olin Starch Formulation #2 - 0.25% HgCl2 as inhibitor	11	11	
133328	100% Cryptomelane (HCl- Lt. Hyd. leach) 7/1 - ore/black	Starch - flour paste 0.25% HgCl2 as inhibitor	11	Polysty- rene cap	
B3332	30% Crypt. (HCl - Lt. Hyd. leach) - 70% Afr. ore - ore/black - 7/1	11	tt	11	
B 3339	30% Crypt. (KCl - Lt. Hyd. leach) - 70% Afr. 7/1 - ore/black	บ	n	11	
B3344	30% Crypt. (NH4Cl - Lt. Hyd. leach) - 70% Afr. 7/1 - ore/black	e s	11	11	
B3547	100% Crypt. (ZnCl2 - Lt. Hyd. leach) 7/1 - ore/black	11	11	11	
B3351	30% Crypt. (2nCl2 - Lt. Hyd. leach) - 70% Afr. 7/1 -ore/black	tı .	11	н	

FIGURE 3 (cont.)

<u> </u>					
	Lot No.	Mix Formulation	Separator	Subse al	Seal
	B3697	100% Crypt. (2nCl2 - western Electro #18 leach) 7/1 - ore/black	Starch - flour paste 0.25% HgCl ₂ as inhibitor	Amprol wax	Polysty- rene cap
	B3701	30% Crypt. (ZnCl2 - W.E. #18 leach) - 70% African 7/1 - ore/black		11	11
	B 3 704	100% Crypt. (NH4Cl - W. E. leach) 7/1 - ore/black	11	11	11
	B3708	30% Crypt. (NH4Cl-W.E. #18 leach) - 70% Afr. 7/1 ore/black	11	11	11
	B4609	30% Crypt. (H01 - Lt. Hyd. leach) - 70% Afr. 7/1 - ore/black		11	11
	B 4 610	30% Crypt. (HCl - Lt. Hyd. leach) - 70% Afr. 7/1 - ore/black	Olin starch formulation #2 - 0.25% HgCl ₂ as inhibitor	11	11 ,
	B4611	30% Crypt. (2nCl2 - Lt. Hyd. leach) - 70% Afr. 7/1 - ore/black	Starch - flour paste 0.25% HgCl ₂ as inhibitor	"	11
-			^		

FIGURE 3 A

	Le	clanche T	ype "D" Ce	lls Stored	nt 113° F	 . (Capacit	у)
				Test	Results		
Lot		BA 30 (da	ys)	BA 8 (ho		BA 408/U	(hours)
No.	Cell No.	Initial	1 yr. at 113°F	Initial	l yr. at 113°F	Initial	l yr. at 113°F
B 31 78	1 2 3	17.0 15.4 17.4	Cells no good	156.0 158.0 160.0	58.7 100.0 70.0	25.0 25.0 25.0	13.5
	Ave	16.6		158.0	76.7	25.0	11.5
B3179	1 2 3	17.0 15.8 17.4	5.0 2.8	208.0 212.0 227.0	Cells no good	25.0 26.0 26.0	Cells no good
	Ave.	16.6	3.9	216.0		25.7	,
B3180	1 2 3	18.2 17.6 16.6	Cells no good	240.0 240.0 240.0	201.6 172.6 184.0	30.0 31.0 30.0	14.3 9.8 13.3
1	Ave.	17.5		240.0	186.1	30.3	12.8
B3328	1 2 3	12.6 13.0 12.6	Cells no good	237.0 244.0 222.0	64.0 54.4	30.0 31.0 27.0	4.0 4.0 4.0
	Ave.	12.7		234.0	59.2	29.3	4.0
B3332	1 2 3	18.2 18.2 17.5	Cells no good	228.0 208.0 220.0	88.0 106.7 77.3	26.0 25.0 27.0	14.7 10.3 13.3
	Ave.	17.9		219.0	90.7	26.0	12.8
B3339	1 2 3	15.8 17.0 18.0	5.6 7.2	215.0 221.0 195.0	134.2 120.0 104.7	27.0 20.0 28.0	16.0 16.7
•	Ave.	16.9	6.4	213.0	119.6	25.0	16.3
B 3 344	1 2 3	16.8 14.8 15.6	6.0 6.2 7.4	168.0 196.0 168.0	131.2 108.0 84.4	19.0 20.0 19.0	10.0 15.0 12.0
le II	Ave.	15.7	6.5	177.0	107.9	19.3	12.3
B3347	1 2 3	15.2 15.6 16.6	Cells no good	167.0 125.0 181.0	60.8 136.0 96.0	29.1 29.5 27.5	11.7 11.6 12.0
	Ave.	15.8		158.0	97.6	28.7	11.8

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FIGURE 3 A (cont.)

	Test Results							
Lot		BA 30 (da	ays)	BA 8 (ho	urs)	BA 408/U	(hours)	
No.	Cell No	Initial	l yr. at 113°F	Initial	1 yr. at 113°F	Initial	l yr. at 113°F	
B3351	1 2 3	16.2 19.2 15.2	8.6 5.6 8.4	199.0 236.0 228.0	85.7 129.5 184.0	27.0 18.0 26.0	11.8 11.7 11.3	
	Ave.	17.5	7.5	221.0	133.1	23.7	11.6	
B3697	1 2 3	23.9 24.4 . 24.4	6.0 5.8	312.0 296.0 296.0	130.0 136.0 83.1	48.0 47.0 48.0	17.3 14.7	
	Ave.	24.2	5.9	301.0	116.4	47.6	16.0	
B3701	1 2 3	18.2 17.2 17.4	8.2 8.0	287.0 268.0 251.0	200.0	26.0 26.0 26.0	15.6 17.4 16.0	
	Ave.	17.6	8.1	269.0	178.2	26.0	16.3	
B3704	1 2 3	9.6 6.6 8.8	4.6	349.0 360.0 344.0	160.0 160.0	32.0 48.0 50.0	Cells no good	
	Ave.	8.3	6.9	351.0	i60.0	43.3		
B3708	1 2 3	19.2 19.6 18.4	7.6 9.0 7.6	272.0 267.0 278.0	129.1 208.0 125.7	29.0 29.0 29.0	15.4 11.6 11.6	
	Ave.	19.1	8.1	272.0	154.2	29.0	12.9	
B4609	1 2 3	15.8 14.4 18.8	7.0 7.4 6.8	216.0 195.0 229.0	102.1 100.8 91.7	27.0 22.0 25.9	9.1 14.3 12.0	
	Ave.	16.3	7.1	213.0	98.2	25.0	11.8	
B4610	2 3	18.8 14.8 15.8	7.0 ' 7.4 6.8	248.0 220.0 224.0	131.2 136.0 96.0	29.1 30.5 28.3	16.0 15.6 16.6	
	Ave.	16.5	7.1	230.0	121.1	29.3	16.1	
B4611	1 2 3	17.6 14.8 15.8	8.4 7.6 8.2	243.0 245.0 244.0	131.6 97.6 136.0	27.7 26.8 27.3	16.0 18.4 12.7	
	Ave.	16.1	8.1	244.0	121.7	27.3	15.7	

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FIGURE 4

LeCla	nche! Type "D" Cells For	Storage At 113° F. (Cons	truction)	
Stora Testi	ge Time Before Initial ng At 70° F.	2 weeks at 70°	F 50% R	•Н•
	ge Time For Cells Stored 3° F. (Tested at 70° F.)	l month at 70° l year at 113°	•	
Lot No.	Mix Formulation	Separator	Subseal	Seal
02327	100% African Ore 7/1 - ore/black 1.0% BaCrO4	Starch - flour paste - 0.30% Na ₂ Cr ₂ O ₇ as inhibitor	Amprol wax	Polysty- rene cap
02330	100% African Ore 7/1 - ore/black	Starch - flour paste - 0.30% Na ₂ Cr ₂ O ₇ as inhibitor	Amprol wax	Polysty- rene cap
02333	100% African Ore 7/1 - ore/black	Starch - flour paste - 0.30% Na ₂ Cr ₂ O ₇ and 0.20% Antaron R155	Amprol wax	Polysty- rene cap
02336	100% African Ore 7/1 - ore/black	Starch - flour paste - 0.20% Antaron R155 as inhibitor	Amprol Wax	Polysty- rene cap
02339	100% African Ore 7/1 - ore/black	Starch - flour paste - HgCl ₂ and 0.20% Ant- aron R155 as inhibitor	Amprol wax	Polysty- rene cap
02342	100% African Ore 7/1 - ore/black	Starch - flour paste - 1.0% Protein R ₂ E-270 without HgCl ₂ inhibite	Amprol wax	Polysty- rene cap
C2345	100% African Ore 7/1 - ore/black	Starch - flour paste- 1.0% Denatured Gluten and no HgCl2 -inhibitor	Amprol wax	Polysty- rene cap
C 23/18	100% African Ore 7/1 - ore/black 1.0% BaCrOL	Starch - flour paste - 0.30% Na ₂ Cr ₂ O ₇ inhibitor graphite treated rods	Amprol wax	Polysty- rene cap
C2351	100% African Ore 7/1 - ore/black 1.0% BaCrOLL	Starch - flour paste- paraffin treated rods	Amprol wax	Polysty- rene cap

FIGURE 4A (Cont.)

LeClanche' Type "D" Cells Stored At 113° F. (Capacity Results)

	1	Type D	Test		s F. (Capa	CILY MESUI	<i>va j</i>
Lot	Aver.	BA-30	(days)	BA-8	(hours)	BA-408/U	(hours)
No.	Cell	Initial	After 1 yr. @113°		After 1 yr. @113		After 1 yr.@113°
02327	Aver.	11.7	9.1	181.6	118.0	23.8	20.7
02330	Aver.	10.2	6.8	180.0	145.0	20.0	16.4
02333	Aver.	10 • 3	5•3	161.2	112.0	22•7	12.6
02336	Aver.	11.3	7•8	175•0	162.0	24.9	9•9
02339	Aver.	12.9	7•2	213.0	155.0	23.1	13.8
02342	Aver.	12.1	9•1	195•0	1 52•0	21.7	13•3
02345	Aver.	12•7	8•3	203.8	171.0	24.5	14.9
02348	Aver.	11.5	7.8	176.0	ग्रे†o•o	25 •6	18.9
02351	Aver.	12•7	7•5	232•5	173.0	23.1	1818

CONSTRUCTION AND CAPACITY RESULTS OF CELLS

STORED AT

130° F.

FOR 3 MONTHS AT 50% R.H.

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FIGURE 5

Leclanche Type "D" Cells For Storage At 130°F (Construction)

Storage time before initial 2 weeks at 70°F, 50% R.H. testing at 70°F							
Storag	Storage time for cells on 130°F 2 weeks at 70°F, 50% R.H. storage (tested at 70°F) 3 months at 130°F, 50% R.H.						
Lot No.	Mix Formulat ion	Separator	Subseal	Seal			
C856	100% African ore 7/1 - ore/black 10% Sal ammoniac	Potato starch, corn starch, flour paste 0.25% HgCl2	Amprol wax	Poly- styrene cap			
C857	100% African ore 7/1 - ore/black 10% Sal ammoniac	Olin starch formulation #2 - 0.25% HgCl2	Amprol wax	Poly- styrene cap			
C858	100% African ore 7/1 - ore/black 20% Sal ammoniac	Potato starch, corn starch, flour paste 0.25% HgCl ₂	Amprol wax	Poly- styrene cap			
C859	100% African ore 7/1 - ore/black 20% Sal ammoniac	Olin Starch formulation #2 - 0.25% HgCl2	Amprol wax	Poly- styrene cap			
0860	30% Lt. Hydrate 70% African ore 7/1 - ore/black	Potato starch, corn starch, flour paste 0.25% HgCl2	Amprol wax	Foly- styrene cap			
C861	30% Lt. Hydrate 70% African ore 7/1 - ore/black	Olin starch formulation #2 - 0.25% HgCl2	Amprol wax	Poly- styrene cap			
C8 62	30% Western Electro ore 70% African ore 7/1 - ore/black	Potato starch, corn starch, flour paste 0.25% HgCl2	Amprol wax	Poly - styrene cap			
0863		Olin starch formulation #2 - 0.25% HgCl2	Amprol wax	Poly- styrene cap			
C8 64	30% Burgess Electro ore 70% African ore 7/1 - ore/black	Potato starch, corn starch, flour paste 0.25% HgCl2	Amprol wax	Poly- styrene cap			
C865	30% Burgess Electro ore 70% African ore .7/1 - ore/black	Olin starch formulation #2 - 0.25% HgCl2	Amprol wax	Foly- styrene cap			
C873	30% Burgess Chem ore 70% African ore 7/1 - ore/black	Potato starch, corn starch, flour paste 0.25% HgCl2	Amprol wax	Poly- styrene cap			

FIGURE 5	
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Lot No.	Mix Formulation	Separator	Subseal	Seal
C874	30% Burgess Chem. ore 70% African ore 7/1 - ore/black	Olin starch formulation #2 - 0.25% HgCl2	Amprol wax	Poly- styrene cap
C875	100% African ore 7/1 - ore/black 10% Sal ammoniac	Corn starch, potato starch, flour paste 0.25% HgCl2	Amprol wax	Poly- styrene cap
C876	100% African ore 7/1ore/black 10% Sal ammoniac	Olin Starch Formulation #2 0.25% HgCl2	Amprol wax	Poly- styrene cap
C8 77	100% African ore 7/1 - ore/black 20% Sal ammoniac	Corn starch, potato starch, flour paste 0.25% HgCl ₂	Amprol wax	Poly- styrene cap
0878 3	100% African ore 7/1 - ore/black 20% Sal ammoniac	Olin starch formulation #2 0.25% HgCl2	Amprol wax	Poly- styrene cap

2

Photone 5A

Leclanche Type "" Cells For atorage at 130° F (Capacity results)

				7, c	st <u>nesults</u>	3		
	Lot		Ba-30 (days)		Bn-8 (hor	irs)	BA-408 (hours)	
	No.	Cell No.	Initial	3 mos. at 150°F	Initial	3 mos. at 130°F	Initial	3 mos. at 130°F
	0856	1 2 3	15.8 16.2 16.8	8.8 7.2 10.0 8.9	227.2 232.0 232.0 250.4	160.0 180.0 104.0	26.8 26.8 25.5 26.3	14.3 13.7 12.0
	0857	Ave.	12.0 12.0 11.8 11.9	4.8 7.2 4.6 5.5	168.0 184.0 168.0	118.0 107.2 92.8 106.0	26.1 25.6 26.0 25.9	13.3 14.7 17.3
	C858	1 2 3	13.8 13.0 13.2	7.8 6.8 8.2	203.4 210.4 219.4	130.8 125.7 118.4	20.0 26.0 26.2 24.1	17.0 17.8 12.0
	C859	. 1 . 2 . 3	13.4 15.4 15.0 15.0	7.6 5.6 2.4 4.8	211.0 200.9 202.0 202.0	124.9 131.2 88.0 106.7	26.1 26.4 26.5	12.0 15.2 11.0
.		Ave.	15.1	4.3	201.8	108.6	26.3	12.7
	C860	1 2 3	19.6 21.4 20.8	8.0 7.2	272.0 267.2 276.8	110.4 134.9	31.7 31.4 30.7	16.0 19.1
		Ave.	20.6	7.6	272.0	122.6	31.3	17.5
	C861	1 2 3	17.0 14.4 16.0	9.4 8.0	242.7 242.3 221.3	110.6 91.2 113.1	29.2 31.7 31.6	10.0 17.6
		Ave.	15.8	' 8.7	255.4	104.9	30.2	13.8
	C862	1 2 3	16.8 17.8 16.6	7.0 6.8 6.4	245.3 250.7 256.0	105.6 116.0 128.0	29.8 28.0 26.4	16.0 15.7 11.8
		AVC.	17.1	6.7	250.4	116.5	28.1	14.5
	C863	1 2 3	11.0	4.6	184.0 184.0	96.0 104.0	26 .7 27 . 1	16.8
		Ave.	11.6	4.6	184.0	100.0	26.9	16.8
	C864	' 1. 2 3	13.6 14.6 13.0	7.6 6.4	266.5 241.5 264.0	117.5 107.2 92.0	26.6 27.4 27.8	18.3 17.0 11.3
·L		Ave.	13.7	7.0	257.5	105.5	27.3	15.5

FIGURE 5A (cont.)

			Test	Results			
Lot	0-13	BA-30 (d	ays)	BA-8 (ho	urs)	BA-408 (hours)	
No.	Cell No.	Initial	3 mos. at 130°F	Initial	3 mos. at 130°F	Initial	3 mos. a
	1	10.0	6.0	178.7	96.0	28.9	13.3
C865	2	9.6		184.0	112.0	28.6	8.0
	3	9.6		178.7	121.6	28.7	19.5
	Ave.	9.7	6 .6	180.5	109.9	28.7	13.2
	1	15.2	7.8	232.0	116.0	25.5	15.5
C873	2	14.2	7.4	224.0	126.8	25.9	17.1
	3	16.2	7.0	232.0	129.5	26.4	15.7
	Ave.	15.2	7.4	229.0	124.1	25.9	16.1
	1	14.0	6.2	178.0	99.3	25.8	17.5
C874	2	10.4	7.4	160.0	126.4	25.2	16.7
	3	11.0		164.0	101.7	25.7	1
	Ave.	11.8	6.8	167.0	109.1	25.5	17.1
	1	16.6	9.0	240.0	127.3	27.8	11.3
C8 75	2	15.4	8.4	244.0	116.8	26.4	15.0
	3	15.6	7.8	245.3	94.9	27.8	14.0
	Ave.	15.9	8.4	243.1	113.0	27.3	13.4
	1	14.0	7.0	184.0	104.0	26.5	16.0
C876	2	11.8	2.8	192.0	92.8	24.0	13.6
0010	3	12.0			72.0	27.5	
	Ave.	12.6	4.9	188.0	89.6	26.0	14.8
	1	16.0	9.0	222.0	125.1	23.6	12.0
	2	16.0	9.0	216.0	130.0	25.5	12.0
	3	16.8	7.6	220.0	122.0	23.2	12.0
	Ave.	16.3	8.5	219.0	125.7	24.1	12.0
C878	1	15.2	9.6	216.0	144.0	24.0	16.0
	2	15.6	6.4	210.0	157.3	24.7	12.0
	3	15.2	8.2	216.0	94.9	24.7	14.5
	Ave.	15.3	8.1	214.0	132.1	24.5	14.2

FI	GURE	6

Leclanche Type "D" Cells Stored At 130°F (Construction)

	ge time before initial ng at 70° F.	2 weeks at 70°F , 50	2 weeks at 70°F , 50% R.H.				
Storag	ge time for cells stored DOF (Tested at 70°F)	2 weeks at 70° F, 50% 3 months at 130° F,	2 weeks at 70°F , 50% R.H. 3 months at 130°F , 50% R.H.				
Lot No.	Mix Formulation	Separator	Subseal	Seal			
C2328	100% African ore 7/1 - ore/black 1.0% BaCrO ₄	Starch - flour paste 0.3% NagCrg07 as inhibitor	Amprol wax	Heat res. polysty- rene cap			
C2331	100% African ore 7/1 - ore/black	п	, 11	, "			
C2334	100% African ore 7/1 - ore/black	Starch - flour paste 0.3% Na ₂ Cr ₂ O ₇ + 0.1% Antaron R155	11	II .			
C2 3 3 7	100% African ore 7/1 - ore/black	Starch - flour paste 0.1% Antaron R155 as inhibitor	11	. 11			
C2 3 40	100% African ce 7/1 - ore/black	Starch - flour paste 0.25% HgCl2 + 0.1% Ant. R155 as inhibitor	n	11			
C2343	100% African ore 7/1 - ore/black	Starch - flour paste 1.0% Protein R2E-570 as inhibitor	11	11			
. C2346	100% African ore 7/1 - ore/black	Starch - flour paste 1.0% Denatured Gluten as inhibitor	a n	11			
C2349	100% African ore 7/1 - ore/black 1.0% BaCrO7	Starch - flour paste 0.3% NagCrgO7 as inhibitor	II .	н			
C2352	100% African ore 7/1 - ore/black 1.0% BaCrO7	Starch - flour paste 0.3% Na2Cr2O7 as inhibitor	11				
C2797	100% A frican ore 7/1 - ore/black	Starch - flour paste 0.25% HgCl2 + 0.1% Ant. R155 as inhibitor	11				

FIGURE 6 A

				Test Re	sults		
Lot		BA 30 (days)		BA 8 (Ho	ours)	BA 408/U (hours)	
No.	Cell No:	Initial	3 mos. at	Initial	3 mos. at	Initial	3 mos. at 130° F
C2328	1 2 3	10.8 10.8 10.4	4.2 7.2 7.2	165.3 166.4 166.7	112.0 117.3 116.4	19.7 19.7 18.9	14.5 11.5 12.8
	Ave.	10.7	6.2	166.1	115.2	19.4	12.9
C2331	1 2 3	9.6 8.8 10.6	3.6 6.6 4.8	160.0 176.0 168.0	84.0 48.0 101.3	21.4 21.5 22.5	11.6 9.0 8.4
	Ave.	9.6	5.0	168.0	77.7	21.8	9.7
C2334	1 2 3	10.4 10.0 10.2	8.2 7.0 7.4	139.0 142.4 142.4	121.6 122.7 136.0	21.2 20.0 20.9	11.0 10.9 10.9
	Ave.	10.2	7.5	141.3	126.7	20.7	10.9
C233 7	1 2 3	10.8 11.2 10.6	6.0 3.6 4.4	156.0 160.0 154.7	90.7 120.0 88.0	18.0 18.8 18.0	11.1 10.4 10.3
	Ave.	10.9	4.6	156.9	99.5	18.3	10.6
C2340	1 2 3	12.6 12.2 12.8	5.4 6.0 5.8	198.4 180.0 184.0	78.4 74.3 90.7	17.7 18.3 17.8	10.7 10.3 10.6
	Ave.	12.5	5.7	187.5	81.1	17.9	10.5
02343	1 2 3	11.6 11.2 11.0	6.2 6.4 4.6	177.7 183.0 191.0	100.6	17.5 17.8 17.2	10.9 10.4 9.8
	Ave.	11.3	5.7	183.9	110.6	17.5	10.3
C2 3 46	1 2 3	12.6 12.4 12.2	5.6 8.6 11.0	192.0 192.0 200.0	168.0 152.0 101.3	22.6 22.0 21.4	12.0 14.1 12.0
	Ave.	12.4	8.4	195.0	140.4	22.0	12.7

FIGURE 6A (cont.)

				Test	Results		
Lot		BA 30 (days)	BA 8 (ho	urs)	BA 408/U	(hours)
No.	No.	Initial	3 mos. at 130°F	Initial	3 mos. at 130° F	Initial	3 mos at
C2349	1 2 3	10.2 10.2 11.2	6.0 8.8 5.2	164.0 165.3 164.0	116.4 118.0 136.0	19.3 18.9 19.5	12.5 14.2 14.6
	Ave.	10.5	6.6	164.4	121.4	19.2	13.7
02352	2 3	12.6 12.0 12.2	8.6 7.2	220.0 204.0 202.7	160.0 148.0 116.8	21.1 22.0 22.3	11.4 11.4 10.4
	Ave.	12.3	7.9	208.9	141.6	21.8	11.0
C2797	2 3	14.6 14.8 14.6	7.8 7.6 7.6	240.0 228.0 232.0	168.0 154.7 145.6	25.5 26.0 25.7	14.0 11.6 15.3
	Ave.	14.6	7.7	233.0	156.1	25.7	13.6

CONSTRUCTION AND CAPACITY RESULTS OF CELLS

STORED AT

160° F.

FOR 2 WEEKS AT 50% R.H.

Security Information
Restricted

FIGURE 7

LeClanche Type "D" Cells For Storage At 160°F (Construction)

	e time before initial g at 70°F		2 weeks at 70°F - 50% R.H.				
Storag storag	e time for cells on 16 e (tested at 70°F)	Oof	2 weeks at 70°F - 50% R.H. 2 weeks at 160°F - 50% R.H.				
Lot No.	Mix Formulation		Separator	Subseal	Seal		
B3160	100% African ore 7/1 - ore/black	0.3%	r - starch paste HgCl ₂ as bitor	amprol wax	polysty- rene cap		
B3161	100% African ore 7/1 - ore/black	0.3%	ial starch paste HgCl ₂ as bitor	amprol wax	polysty- rene cap		
B3162	100% African ore 7/1 - ore/black 1.0% BaCrO4	0.3%	r - starch paste Na ₂ Cr ₂ O ₇ as bitor	amprol wax	polysty- rene cap		
B3163	100% African ore 7/1 ore/black 1.0% BaCrO4	0.3%	ial starch paste Na2Cr2O7. as bitor	amprol wax	polysty- rene cap		
B3164	100% African ore 7/1 - ore/black 0.5% Cr03	0.3%	r - starch paste Na ₂ Cr ₂ O ₇ as bitor	amprol wax	polysty- rene cap		
B3165	100% African ore 7/1 - ore/black 0.5% Cr03	0.3%	ial starch paste NagCrgO7 as bitor	amprol wax	polysty- rene cap		
B3166	100% African ore 7/1 ore/black 0.5% Cr03	0.3%	r - starch paste Na2Cr2O7 as bitor	amprol wax	polysty- rene cap		
B3167	100% African ore 7/1 - ore/black 0.5% Cr03	Cron	ial starch paste aked cans Na2Cr2O7	amprol wax	polysty- rene cap		

FIGURE 7 (cont.)

Lot No.	Mix Formulation	Separator	Subseal	Seal
B31 68	100% African ore 7/1 - ore/black 1.0% BaCrO4	Flour - starch paste Cronaked cans 0.3% Na2Cr2O7	amprol wax	polysty- rene cap
B3169	100% African ore 7/1 - ore/black 1.0% BaCrO4	Special starch paste Cronaked cans 0.3% Na ₂ Cr ₂ O ₇	amprol wax	polysty- rene cap
B3170	100% African ore 7/1 - ore/black	Special starch - flou paste - 0.3% Na2Cr297 as inhibitor		polysty- rene cap
B3171	100% African ore 7/1 - ore/black	Special starch-flour paste - 0.3% HgCl2 as inhibitor	amprol wax	polysty- rene cap
B3173	100% African ore 7/1 - ore/black 1.0% BaCr04	Special starch paste 0.3% Na2Cr2O7 as inhibitor	emprol wax	polysty- rene cap
B3174	100% Western Electro ore - 7/1 ore/black	Special starch paste. 0.3% HgCl2 as inhibitor	amprol wax	polysty- rene cap
B3175	50% Western Electro 50% African ore 7/1 - ore/black	Special starch paste 0.3% HgCl2 as inhibitor	amprol wax	polysty- rene cap
B3176	33% Light Hydrate 67% African ore 7/1 - ore/black	Special starch paste 0.3% HgCl ₂ as inhibitor	amprol wax	polysty- rene cap
B3177	100% African ore 7/1 - ore/black	Starch - flour paste 0.3% HgCl2 as inhibitor	amprol wax	hard wax
B3327	100% Crypt. (H20 4 Lt. Hyd.) 7/1 - ore/black	Starch - flour paste 0.3% HgCl2 as inhibitor	amprol wax	polysty- rene cap

FIGURE 7 (cont.)

Lot No.	Mix Formulation	Separator	Subseal	Seal
B3330	30% Cryptomelane 70% African ore 7/1 - ore/black	Starch - flour paste 0.3% HgCl2 as inhibitor	amprol wax	polysty- rene cap
B3334	100% Crypt. (KC1 - Lt. Hyd.) 7/1 - ore/black	Starch - flour paste 0.3% HgCl ₂ as inhibitor	amprol wax	polysty- rene cap
B3337	30% Cryptomelane 70% African ore 7/1 - ore/black	Starch - flour paste 0.3% HgCl ₂ as inhibitor	amprol wax	polysty- rene cap
B3342	30% Crypt. (NH4Cl - Lt. Hyd.) 70% Afr. ore 7/l - ore/black	Starch - flour paste 0.3% HgCl2 as inhibitor	amprol wax	polysty- rene cap
B3346	100% Crypt. (ZnCl2 - Lt. Hyd.) 7/1 - ore/black	Starch - flour paste 0.3% HgCl2 as inhibitor	amprol wax	polysty- rene cap
B3349	30% Cryptomelane 70% African ore 7/1 - ore/black	Starch - flour paste 0.3% HgCl2 as inhibitor	amprol wax	polysty- rene cap
B3696	100% Crypt. (ZnCl2 - W.E. ore) 7/1 - ore/black	Starch - flour paste 0.3% HgCl ₂ as inhibitor	amprol wax	polysty- réne cap
B3699	30% Cryptomelane 7 0% African ore 7/1 - ore/black	Starch - flour paste 0.3% HgCl2 as inhibitor	amprol wax	polysty- rene cap
B3703	100% Crypt. (NH4Cl - Western Electro ore) 7/1 - ore/black	Starch - flour paste 0.3% HgCl2 as inhibitor	amprol wax	polysty- rene cap
B3706	30% Cryptomelane 70% African ore 7/1 - ore/black	Starch - flour paste 0.3% HgCl2 as inhibitor	amprol wax	polysty- rene cap

FIGURE 7 (cont.)

Lot No.	Mix Formulation	Separator	Subseal	Seal
B4582	50% Crypt. (NH4Cl - W.E. ore) - 50% A fr. ore 7/l - ore/black	Starch - flour paste 0.3% HgCl2 as inhibitor	amprol wax	polysty- rene cap
B4737	100% African ore 7/1 - ore/black	Starch - flour paste 0.3% HgCl ₂ as inhibitor	amprol wax	polysty- rene cap
B4846	30% Crypt. (ZnCl2 - W.E. ore) - 70% Afr. ore - 7/1 - ore/black	Starch - flour paste 0.3% HgCl ₂ as inhibitor	amprol wax	polysty- rene cap
B 4911	30% Western Electro 70% African ore 7/1 - ore/black	Starch - flour paste 0.3% HgCl ₂ as inhibitor	amprol wax	polysty- rene wax
C787	100% African ore 7/1 - ore/black	Starch - flour paste 0.3% HgCl ₂ as inhibitor	amprol wax	polysty- rene cap
C788	100% African ore 7/1 - ore/black	Special starch paste 0.3% HgCl ₂ as inhibitor	amprol wax	polysty- rene cap
B 473 8	100% African ore 7/1 - ore/black	Starch - flour paste 0.3% HgCl2 as inhibitor	amprol wax	polysty- rene cap
B4739	100% African ore 7/1 - ore/black	Special starch paste 0.3% HgCl2 as inhibitor	amprol wax	polysty- rene cap
B4847	30% Crypt. (ZnCl2 - W.E. ore) 70% African Ore - 7/1 - ore/black	Starch - flour paste 0.3% HgCl2 as inhibitor	amprol wax	polysty- rene cap
C1 623	100% African ore 7/1 - ore/black	Starch - flour paste 0.3% HgCl2 as inhibitor	amprol wax	polysty- rene cap

FIGURE 7A

LeClanche Type "D" Cells For Storage At 160°F (Capacity)

			T				o cer	Ta F,	or Sto	rage	At 16	0°F'	(Capac	1ty)		
		ot o.		Cell	BA-	30 (days)		Test		sults					
	-	·	1	10.	Initi	.al	2 wks	. at	Init		2 wks	. at			/U (hou	
	В3	160	- :		13. 13. 14.(4	5.8 6.4 5.8 5.2		220 178 190 190	3)	101 107 113	F.	Init 19, 18, 20.	0	2 wks 160°	F. 0
			Av	θ. ·	13.8		5.8				97 113				10.0 10.0 11.0)
- 1			1		14.2				195		106		19.	0	10.6	
.	B31	61	3 4		13.4		2.4		17 8 192 192	3	58.0 79 60 60	o	19.0 20.0 19.0		3.0 6.0 9.0	
-		-	Ave	-	13.9		2.4		187	+					1.0	
		- 1	1		11.2					+	64.2		19.3		4.8	_
I	B316		2 3 4 5		11.0		4.6 5.2 5.6 3.2 2.0		162 172 168 168		24.0 64.0 80.9 61.0		21.0 21.0 21.0 22.0		13.0 9.0 9.0 10.0	
-		- A	ve.		11.2		4.1	+	160							
			1		11.2	ŀ	2.6	1	168	+-	58.0	-	21.3	-	10.3	1
B3	3163	1 8			11.4		2.6 4.8 5.0 2.2		157 166		129.0 120.0 120.0 144.0 .16.0	dis thr wro	ng		12.0 10.0 10.0	
_		AV	0.	1	1.4		3.4	. 1	161			-	1	1	0.0	
B31	L64 ·	2 3 4		1:	1.6	no	lls od	1	.68 68 68	(26.0 30.0 58.0 40.0	2	2.0	. e	0.4 3.0 3.0	,
		Ave	•	11	.6			16	88						1	,
•							-		, <u>u</u>	5	9,5	2]	.7	8	.0	

FIGURE 7A (cont.)

			ſ	Test Resu	lts		
Lot		BA-30 (days)	BA-8 (h	ours)	BA-408/	U (hours)
No.	Cell No.	Initial	2 wks. at 160° F.	Initial	2 wks. at 160° F.	Initial	2 wks at 160° F.
B3165	1 2 3 4 5	12.4 12.2 12.2	3.0 1.0 2.4 4.0	166 168 166	77.0 99.0 99.0 102.0 108.0	23.0 23.0 23.0	11.0 10.0 11.0 9.0 11.0
	Ave.	12.3	2.6	166.7	97.0	23.0	10.4
B3 1 66	1 2 3 . 4	12.2 13.6 13.0	Cells no good	156 165	53.3 64.0 15.0 44.0	23.0 23.0 23.0	4.0
	Ave.	12.9		160	44.1	23.0	4.0
B3167	1 2 3 4 5	11.0 12.0 11.8	2.2 2.0 3.8 3.0 1.3	177 179 177	81.7 99.7 89.0 45.3 88.2	25.0 25.0 26.0	10.8 7.1 9.7 11.5
	Ave.	11.6	2:.5	17 8	81.0	25.3	9.8
B3168	1 2 3 4	12.8 12.2 12.4	2.0 7.2 7.6	202 197 211	88.0 47.0 88.0 107.0	25.0 25.0 25.0	9•8 9•6
	Ave.	12.5	7.4	203	82.5	25.0	9•7
B3169	1 2 3 4 5	12.6 13.4 12.6 11.6	4.0 5.0 5.0 5.0 2.0	198 189 192 192	109.7 88.0 54.4 136.7 48.0	25.0 25.0 26.0 25.0	3.0 4.0 8.0 10.0
	Ave.	12.6	4.3	193	88.0	25.2	6.3

FIGURE 7 A (cont.)

			Te	est Resul	ts		
Lot	Cell	BA-30 (days)	BA-8 (h	ours)	BA-408/U (hours)	
йо•	No.	Initial	2 wks at 160° F.	Initial	2 wks at 1600 F.	Initial	2 wks at 160° F.
B3170	1 2 3 4 5	11.6 12.4 12.6 12.4	2.2 4.4 4.2	181.3 184.0 184.0 184.0	56.0 90.7 101.3 54.3 40.0	25.5 25.5 25.5	4.0 9.0 9.0
	Ave.	12.3	3.6	183.3	68.5	25.5	7.3
B3171	1 2 3	16.2 18.0 17.8	3.0 7.4	220.0 222.0 220.0	88.0 88.0	29.0 27.0 25.0	Cells no good
	4	17.3	5.2	221.0	88.0	27.0	
B3 17 3	1 2 3	13.0 11.6 12.2	Cells no good	188.0 185.0 184.0	48.0 61.3 37.3	NO results	Cells no good
	Ave.	12.3		186.0	49.0		
B3174	1 2 3	24.8 24.6 24.8	5.2 .3.2	324.0 293.0 264.0	152.0	45.0 47.0 46.0	Cells no good
	Ave.	24.7	4.2	294.0	152.0	46.0	200 May 200, 200
B3175	1 2 3 4	15.0 14.2	Cells no good	252.0 230.0 249.0	64.0 116.0 88.0 120.0	31.0 32.0 31.0	16.0 12.0 17.0
	A v e.	14.6		244.0	90.4	31.3	15.0

· FIGURE 7 A (cont.)

				Test Resu	ılts			
Lot	Cell	BA-30 (d	ays)	BA-8 (ho	ours)	BA-408/U (hours)		
No.	No.	Initial	2 wks at 1600 F.	Initial	2 wks at 160° F.	Initial	2 wks at 160° F.	
B3176	1 2 3 4	20.6 17.0 19.4 19.4	6.0	233.0 235.0 228.0 239.0	53.3 24.0 42.7 87.8	32.0 31.7 31.7 30.2	6.0	
	Ave.	19.1	6.0	234.0	52.0	31.4	6.0	
B3177	1 2 3 4	No results	Cells no good	162.0 206.0 240.0 184.0	24.0 50.0 96.0 23.0	No results	Cells no good	
	Ave.			198.0	56.3			
B3327	1 2 3 4	9.6 11.6 12.0	1.4	210.0 212.0 225.0	54.4 56.0 58.7	21.0 22.0 22.0 21.0	4.0 4.0 6.0	
	Ave.	11.1	1.5	216.0	. 56.4	21.5	4.7	
B3330	1 2 3	13.2 13.6	Cells no good	182.0 165.0 172.0	2.1 51.4 44.9	17.5 18.6 16.9	1.0	
	Ave.	13.4		173.0	48.1	17.7	1.5	
B3334	1 2 3 4	9.4 11.0 9.2	7.4	193.0 193.0 182.0	35.0 24.0 29.0	23.0 21.0 20.0 24.0	Cells no good	
	Ave.	9.9	7.4	189.0	29.3	22.0	the two feet day 1/4	
B3337	1 2 3 4	13.0 13.2 14.2	Cells no good	193.0 183.0 192.0	35.0 40.0 12.0	19.0 23.0 24.0 23.0	4.0	
	Ave.	13.5		190.0	37.5	22.3	4.0	

FIGURE 7A (cont.)

				Test Re	esults			
Lot	Cell	BA-30 (d	ays)	BA-8 (he	ours)	BA-408/U (hours)		
No.	No.	Initial	2 wks. at 160° F.	Initial	2 wks at 160° F.	Initial	2 wks at 1600 F.	
B3342	1 2 3 4	12.6 10.0 14.2	3.4 2.2 3.6	115.0 132.0 128.0	52.6 58.0 72.0	16.0 14.0 15.0 16.0	8.0 7.0 5.0 8.0	
	Ave.	12.3	3.1	125.0	60.9	15.3	7.0	
B3346	1 2 3 4	12.6 14.0 12.8	5.4 4.8 2.8	171.0 174.0 164.0	83.0 88.0 64.0	26.0 26.0 26.0 26.0	9.0 9.0 11.0 10.0	
	Ave.	13.1	4.3	170.0	78.0	26.0	9.0	
B3349	1 2 3 4	15.8 14.6 14.6	6.8 6.0 6.4	195.0 205.0 195.0	81.0 77.0 78.0	16.4 15.8 16.9 17.3	9.0 9.0 10.0 10.0	
	Ave.	15.0	6.4	198.0	79.0	16.6	9.5	
B3696	1 2 3 4	22.2 20.8 21.2	Cells no good	289.0 273.0 299.0	108.0 42.0 50.0	36.0 39.0 39.0 39.0	Cells no good	
	Ave.	21.4		287.0	67.0	38.2		
B3699	2 3 4	16.0 17.4 14.8	6.8 7.6 7.2	232 240 240	113.0 142.0 130.0	24.0 23.0 23.0 23.0	14.5 14.0 15.1 15.4	
	Ave.	16.1	7.2	237.0	128.0	23.2	14.7	
B3703	1 2 3 4	4.8 6.4 4.2	Cells no good	243.0 264.0 324.0	88.0	10.0 15.0 12.0 13.0	Cells no good	
	Ave.	5.1		277.0	88.0	12.5		

FIGURE 7 A (cont.)

				Test Re	sults		
Lot No.	Ce11	BA-30 (days)	BA-8 (h	ours)	BA-408/U	(hours)
	No.	Initial	2 wks at 160° F.	Initial	2 wks at 160° F.	Initial	2 wks at 160° F.
B3706	1 2 3 4	17.4 17.6 19.2	8.0 7.0 8.6	229.0 233.0 256.0	144.0 131.0 108.0	25.0 26.0 26.0	13.0 14.0 13.0 13.0
	Ave.	18.1	7.9	239.0	128.0	25.7	13.2
B4582	1 2 3	15.6 15.6 14.2	4.4 4.6 5.2	202.0 208.0 228.0	84.0 98.7 26.9	24.4 26.6 20.0	7.2 5.5 8.0
	Ave.	15.1	4.7	213.0	91.3	23.7	6.9
B4737	1 2 3	12.6 13.2 13.8	6.0 6.2 6.1	208.0 190.0 186.7	105.0 109.0 96.0	20.6 21.3 21.4	10.3 10.7 10.3
	Ave.	13.2	6.1,	195.0	104.0	21.1	10.4
B 4 846	1 2 3 4	15.6 14.8 13.8 14.6	7.0 7.0 6.8 3.6	256.0 238.4 235.2 250.7	94.0 124.0 133.6 126.1	26.5 26.8 24.9 26.2	14.3 16.0 13.8 15.7
· · · · · · · · · · · · · · · · · · ·	Ave.	14.7	6.1	245.0	117.0	26.1	14.9
B 4911	1 2 3	16.8 17.6 15.2	8.2 7.3 6.0	192.0 204.0 164.0	112.4 126.9 85.3	20.0 26.2 26.2	13.0 17.0 18.0
	Ave.	16.5	7.1	187.0	108.0	24.1	16.0
0787	1 2 3	13.6 12.8 12.8	2.8	192.0 194.0 194.0	72.8	18.9 18.2 18.7	4.0
	Ave.	13.1	2.8	193.0	72.8	18.6	4.0
0788	1 ° 2 3	13.6 13.6 13.0	4.0 6.0 2.8	176.0 186.0 186.0	84.4 77.3 83.2	19.4 19.5 19.5	10.2 9.6 4.0
,	Ave.	13.4	4.3	183.0	80.6	19.5	9.9

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FIGURE 7A (cont.)

				lest Resul	ts		,
Lot	Cell	BA-30 (d	ays)	BA-8 (ho	urs)	BA-408/U (hours)	
No.	No.	Initial	2 wks at 160° F	Initial	2 wks at 160° F	Initial	2 wks at 160° F
B4738	1 2 3	12.8 12.6 12.4	5.0 5.4 5.2	196.0 201.0 208.0	96.0 114.7 82.0	21.8 21.7 21.4	10.9 10.9 10.3
	Ave.	12.6	5.2	202.0	97.6	21.6	10.7
B4739	1 2 3	12.4 13.0 12.5	3.2 6.4 6.0	198.4 192.0 185.8	118.0 96.0 99.3	23.3 21.9 22.9	wrong discharge
	Ave.	12.6	5.2	192.0	107.8	22.7	
B4847	1 2 3	13.6 14.4 15.6	5.6 5.2 4.2	232.0 250.7 226.7	112.0 107.6 94.9	27.0 26.5 27.2	8.0 13.0 11.2
	Ave.	14.5	5.0	237.0	104.8	26.9	10.7
C1623	1 2 3	14.0 15.0 13.4	9.8 8.8 8.4	220.0 212.0 236.0	155.1 176.0 136.0	18.5 25.1 22.0	11.8 11.6 11.6
	Ave.	14.1	9.0	223.0	155.7	21.9	11:.7

FIGURE 8

	### V	FIGURE 8	,	
	Leclanche Type "D"	Cells Stored At 160° F (Constructi	.on)
Stora testi	ge time before initial ng at 70° F	2 weeks at 70° F , 50)% н. н.	
Stora at 16	ge time for cells stored 0°F (tested at 70°F)	2 weeks at 70° F , 50 2 weeks at 160° F, 50	0% R.H. 0% R.H.	
Lot No.	Mix Formulation	Separator	Subseal	Seal
C23 2 9	100% African ore 7/1 - ore/black 1.0% BaCrO4	Starch - flour paste 0.3% NagCrg07 as inhibitor	Amprol wax	Heat res. polysty- rene cap
C2332	100% African ore 7/1 - ore/black	t1	11	II
C2 3 35	100% African ore 7/1 - ore/black	Starch - flour paste 0.3% Na2Cr207 + 0.1% Antaron R 155	II.	11
C 233 8	100% African ore 7/1 - ore/black	Starch - flour paste 0.1% Antaron R155 as inhibitor	11	11
C2341	100% African ore 7/1 - ore/black	Starch - flour paste 0.25% HgCl2 + 0.1% Antaron R155	II	н
C2344	100% African ore 7/1 - ore/black	Starch - flour paste 1.0% Protein R2E-570 as inhibitor	11	19
C2347	100% African ore 7/1 - ore/black	Starch - flour paste 1.0% Denatured Gluten as inhibitor	11	п
02350	100% African ore 7/1 - ore/black	Starch - flour paste 0.3% Na2Cr2O7 as inhibitor	11	11
C2353	100% African ore 7/1 - ore/black		. 11	ll ll
C2798	100% African cre 7/1 - ore/black	Starch - flour paste 0.20% HgCl2 + 0.1% Ant. as inhibitor	Polyethyl Gasket no wax subseal	11

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FIGURE 8 (cont.)

Lot No.	Mix Formulation	Separator	Subseal	Seal
C2774	100% African ore 7/1 - ore/black	Starch - flour paste 0.25% HgCl2 as inhibitor	Polyeth- ylene gasket - no wax	Heat res. polysty- rene cap
C2775	100% African ore 7/1 - ore/black	11	Ħ	n
C2776	100% African ore 7/1 - ore/black	11	Polyethyl gasket + amprol wax	11
C3267	100% Cryptolite 7/1 - ore/black	"	polyethylene gasket - no wax	н
C3268	100% Cryptolite 7/1 - ore/black	Olin starch formulation #2 - 0.25% HgCl2 as inhibitor	11	Ħ
03271	30% Cryptolite - 70% African ore 7/1 - ore/black	Starch - flour paste 0.25% HgCl2 as inhibitor	H	11
C 3272	30% Cryptolite - 70% African ore 7/1 - ore/black	Olin starch formulation #2 - 0.25% HgCl2 as inhibitor	11	n .
03275	100% Anodite 7/1 - ore/black	Starch - flour paste 0.25% HgCl ₂ as inhibitor	Ħ	10
C3276	100% Anodite 7/1 - ore/black	Olin Starch formulation #2 - 0.25% HgCl2 as inhibitor	11	H.
C3279	30% Anodite - 70% African ore 7/1 - ore/black	Starch - flour paste 0.25% HgCl2 as inhibitor	11	11.
03280	30% Anodite - 70% African ore 7/1 - ore/black	Olin starch formulation #2 - 0.25% HgCl ₂ as inhibitor	11	H

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FIGURE 8A

Leclanche Type "D" Cells Stored At 160° F (Cap	pacity)
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				Test	Results		
Lot	Cell No.	BA 30 (days)		BA 8 (hours)		BA 408/U (hours)	
No.		Initial	2 wks. at 160°F	Initial	2 wks. at 160° F	Initial	2 wks. at
C2329	1 2 3	11.6 10.0 10.8	8.8	157.7 168.0 166.4	16.0 25.5	19.7 19.3 19.5	14.3
	Ave.	10.8	8.8	164.0	20.7	19.5	14.3
C2332	1 2 3	9.6 10.0 9.6	6.0 5.6 5.4	168.0 160.0 160.0	107.6 108.0 112.0	18.8 18.9 24.5	10.8 11.7 11.8
	Ave.	9.7	5 .7	162.7	109.2	20.7	11.4
C2335	1 2 3	10.2 10.2 10.4	7.4 9.0 8.0	165.3 158.0 165.3	133.3 133.3 131.2	18.3 19.0 19.3	13.0 12.0 12.0
	Ave.	10.3	8.1	162.9	132.6	18.8	12.3
C2338	2 5	10.0 10.2 10.2	5.8 6.4 6.8	156.8 158.0 155.4	86.6 100.9 120.0	21.1 20.0 20.2	11.4 9.8 12.0
	Ave.	10.2	6.3	156.7	102.5	20.4	11.1
C 2341	1 2 3	11.8 12.6 11.8	8.0 3.2	184.0 188.0 196.0	112.0 105.1 68.8	18.7 16.7 18.2	9.4 10.8 9.8
	Ave.	12.1	5.6	189.0	95.3	17.8	10.0
C2344	2 3	11.2 11.8 11.4	6.6 5.8 8.0	178.7 192.0 176.0	112.0 100.0 108.8	18.5 16.8 17.4	10.4 10.2 10.6
	Ave.	11.5	6.8	182.2	106.9	17.6	10.4
C2 347	1 2 3	11.8 12.8 11.8	1.6 3.6 1.0	180.0 184.0 168.0	64.0 108.0 92.0	17.7 18.3 19.7	10.0 7.6 4.0
, 	Ave.	12.1	2.1	177.0	88.0	18.5	7.2

FIGURE 8A (cont.)

				Test	Results		
Lot No.	Cell No.	BA 30 (days)		BA 8 (hours)		BA 408/U (heurs)	
		Initial	2 wks. at 160°F	Initial	2 wks. at 160°F	Initial	2 wks. at 160°F
C2350	2 3	10.4 10.4 11.0	9.4 9.6 10.2	166.0 156.0 168.0	21.3	20.0 19.0 19.1	6.9 4.0
	Ave.	10.6	9.7	163.0	21.3	19.3	5.4
C2353	1 2 3	11.2 11.8 11.8	4.6 8.2 4.6	216.0 212.0 214.0	136.0 144.0 144.0	22.4 16.7 18.4	11.7 12.0 10.7
	Ave.	11.6	5.8	214.0	141.3	19.2	11.4
C2798	1 2 3	14.2 12.8 14.2	7.0 8.0	244.0 242.7 245.3	142.0	25.3 20.0 22.7	12.8 10.5
	Ave.	13.7	5.7	244.0	143.0	22.6	11.7
C2774	1 2 3	13.8 16.8 16.2	6.6 6.6 8.8	232.0 221.3 236.0	136.0 160.0 112.0	25.5 24.7 25.4	11.0 11.5 13.1
	Ave.	15.6	7.3	229.8	136.0	25.2	11.8
02 775	1 2 3	14.2 12.8 14.2	9.6 11.4 12.0	244.0 242.7 245.3	164.0 145.2 197.3	25.3 20.0 22.7	18.0 18.9 16.0
	Ave.	13.7	11.0	244.0	168.8	22.6	17.8
C2776	1 2 3	14.2 12.8 14.2	12.4 11.2 11.2	244.0 242.7 245.3	196.0 197.3 168.4	25.3 20.0 22.7	18.0 16.0 17.1
	Ave.	13.7	11.5	244.0	180.4	22.6	17.1
C3267	1 2 3	22.8 18.6 19.4	8.0 6.2 8.0	328.0 328.0 332.0	94.9 128.0 82.7	28.4 40.0 39.0	12.0 15.7 14.7
	Ave.	20.3	7.5	329.0	101.8	35.8	14.1

FIGURE 8A (cont.)

				Test	Results		
Lot No.	Cell	BA 30	days)	BA 8 (h	ours)	BA 408/U	(hours)
		Initial	2 Wks. at 160°F	Initial	2 wks. at 160°F	Initial	2 wks. at
C3268	1 2 3	21.6 22.4 22.6	8.6 8.6 9.2	328.0 336.0 256.0	117.5 109.3 96.0	34.8 40.0 39.5	24.0 26.2 25.5
	Ave.	22.2	8.8	306.0	107.6	38.1	25.2
C3271	1 2 3	17.2 16.6 17.0	2.0 1.2 1.0	256.0 2 39.1 20 3. 0	97.0 114.2 99.1	29.6 27.3 28.7	14.3
	Ave.	16.9	1.4	238.3	103.4	27.5	14.3
C3272	2 3.	12.2 13.0 8.8	6.0 6.8 7.8	197.1 188.0 216.0	136.0 130.7 136.0	26.9 28.7 30.8	10.7
	Ave.	11.3	6.9	200.3	134.2	28.8	9.9
C3275	1 2 3	21.6 17.2 20.0	3.8 ,	272.0 296.0 280.0	Cells no good	32.8 33.2 30.7	Cells no good
	Ave.	19.6	3.8	289.3		32.2	
C3276	1 2 3	12.8 12.8 12.8	4.8 6.0 4.2	272.0 196.0 216.0	116.8 16.0 42.7	35.2 32.8 36.0	4.0
	Ave.	12.8	5.0	228.0	58.5	34.7	5.8
C3279	1 2 3	15.6 15.6 14.6	9.2 10.0 9.6	232.0 221.3 236.0	117.7 105.2 145.1	25.6 25.2 26.4	15.2
	Ave.	15.3	9.6	229.8	122.7	25.7	15.8
C3280	1 2 3	12.6 12.8 13.6	3.2	205.3 202.7 204.0	94.0 136.0 109.7	27.0 27.4 27.8	12.0 12.0 10.5
	Ave	13.0	3.2	204.0	113.2	27.4	11.5

RECOMMENDATIONS

- 1. The designed polarization apparatus be employed to evaluate new anodic inhibitors eliminating the complicated reactions occuring in the cathode area.
- 2. Manganese dioxides having a stable crystal structure and giving consistently high capacity results should be used in the fabrication of cells for storage condition specified in this contract. Gamma-B and cryptomelane crystal structures afford the best characteristics in achieving this purpose.
- 3. Paper lined cells using Eveready methyl cellulose coated paper, African ore depolarizer, polyethylene gasket and heat resistant polystryene top seal, will meet the storage conditions of 130° F, 50% R.H. and 160° F, 50% R.H.
- 4. Polyethylene gasket substituted for the wax sub-seal and heat resistant polstyrene top seal will produce an effective seal that is resistant to cell leakage.
- 5. The application of (0.3%) ammonium chromate inhibitor (by weight of electrolyte in the separator) and 1% barium chromate (reservoir in the mix) for fabricated cells create performances which meet the conditions specified by this contract.